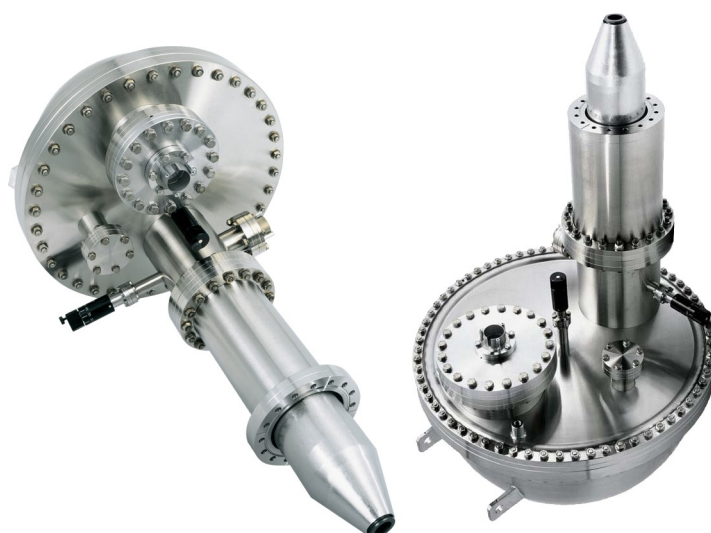


Surface Analysis
Technology

Vacuum
Components



Surface Analysis Computer
System Software Technology



PHOIBOS

Hemispherical Energy Analyzer Series

PHOIBOS 100 / PHOIBOS 150

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User manual for the Hemispherical Energy Analyzer Series **PHOIBOS 100/150**.

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**SPECS GmbH - Surface Analysis
and Computer Technology
Voltastrasse 5
13355 Berlin
GERMANY
PHONE: +49 (0)30 46 78 24 -0
FAX: +49(0)30 4 64 20 83
<http://www.specs.de>**

Chapter

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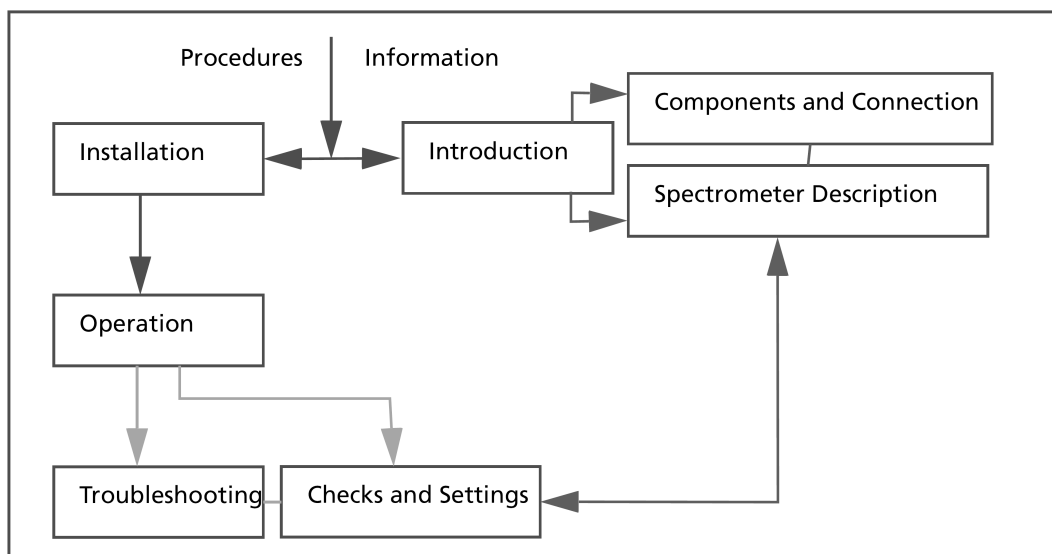
Chapter

1

Introduction

1.1 Overview

Chapters in this manual:



The SPECS PHOIBOS hemispherical electrostatic energy analyzer allows recording of energy spectra for negative particles (electrons) and positive particles (ions) in the kinetic energy range from 0 eV to 3.5 keV.

The PHOIBOS series of hemispherical analyzers are hemispherical deflectors available in two sizes, 100mm or 150 mm mean radii. The input lens is designed to accommodate a wide range of applications.

The analyzer can be equipped with single-channel and optional multichannel detectors; a 5-channel detector for the PHOIBOS 100 and a 9-channel detector for the PHOIBOS 150. The detection electronics incorporates a discriminator, preamplifier, counter and

bus interface. All parts are integrated into a single, compact RF-shielded aluminum case. The detection electronics are supplied together with the analyzer control unit.

PHOIBOS analyzers provide the detection of electron and ion energies between 0 and 3500 eV with minimum step widths of 7 meV. For ultra high energy resolution applications the unit can be operated in a 400 V or a 40 V range with extremely low ripple. Step widths down to 80 μ V are possible in the lowest voltage range.

A multi-element, two-stage transfer lens can be operated in several different modes for angular and spatially resolved studies. All lens modes can be set electronically. A Slit-Orbit mechanism and a Multi-Mode Lens make the sampling area of the analyzer and the acceptance angle area of the lens selectable. Thus the analyzer allows spatially resolved measurements down to a diameter of 100 μ m as well as large area investigations associated with different lens acceptance angles.

All units are completely controlled by SPECS software. Operation of the software will be described in a separate manual.

Typical uses of the PHOIBOS analyzer include photoelectron spectroscopy (XPS, SSXPS, UPS), Auger electron spectroscopy (AES, SAM) and ion scattering spectroscopy (ISS). The PHOIBOS analyzer is bakeable up to 200°C after removal of the detector electronics and the connection for the lens supply.

Safety Information

**Before any electric or electronic operations please consult
"SPECS Safety Instructions" and follow them strictly.**

Some adjustments that have to be performed in this manual are dangerous. At each point these are indicated by a warning label:



Warning!

Tests to be performed on the electronic unit are with its cover removed. Hazardous voltages are present. Only trained, qualified personnel are allowed to perform this task.

Chapter

2

Components and Connections

2.1 System Description, Package Contents

The contents of your system should include the components listed below. Please refer to figure 1 for photos of these.

- PHOIBOS analyzer
 - The PHOIBOS analyzer consists of the following internal parts:
 - analyzer housing,
 - internal μ -metal shielding,
 - lens system,
 - hemispherical analyzer,
 - multichannel (MCD) or single detector (SCD)
 - Slit Orbit mechanism.
- HSA3500 power supply for PHOIBOS analyzer (fully remote controlled).
- and the following additional parts:
 1. Mounting instructions
 2. Manual Safety Instructions
 3. Manual PHOIBOS
 4. Manual SpecsLab2
 5. Manual CasaXPS
 6. Second network card (Fast Ethernet PCI Adaptor)
 7. Detector electronics PCU 300
 8. Handle for power supply HSA3500
 9. 2 x RS232 cables - CAN bus cable
 10. Pair of DN100CF copper gaskets
 11. Cross over cable 2m
 12. Ethernet cable TP 2m
 13. 2 x terminator plugs CAN bus
 14. Power line cables
 15. EC10 CAN-Ethernet-Adapter (section on page)
 16. Magnet holder for EC10 Adapter
 17. Vacuum interlock plug (shorten pin1-pin2)
 18. HSA3500 cabinet feet

19. Analyzer box with filter unit (cube or cylinder shape) , fixed cable
20. SHV-cable ChannelBase
21. SHV-cable ChannelHV
22. SpecsLab2 Installation CD
23. Specification (Test Report)
24. Pair of DN40CF copper gaskets
25. Pair of DN16CF copper gaskets
26. Two Lugs for analyzer lift
27. Two Lugs for analyzer support (only PHOIBOS 150)
28. Screws for lens flange
(screws and nuts for PHOIBOS 100 and bolts and nuts for PHOIBOS 150)

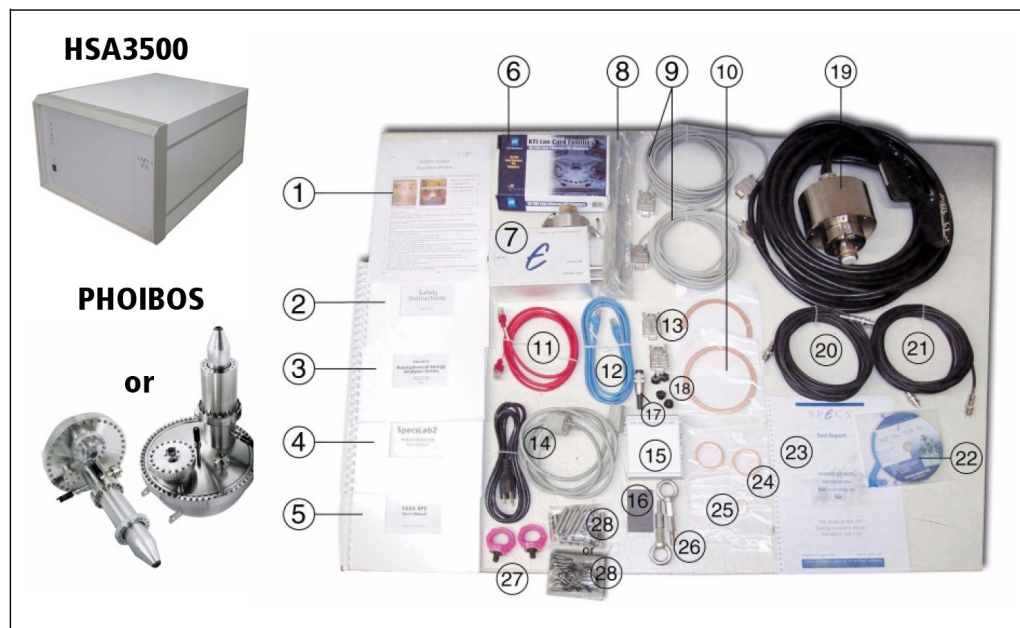


Figure 1: Package Contents

2.2 Electrical Connections

The electrical connection diagram is shown in figure 2, page 7. The connection to the analyzer and the detector are supplied by two multi-pin vacuum feedthroughs that are designed for high voltages up to 5 kV. More detailed descriptions are given in section and section 8.4 .

All devices must be switched off before connecting or removing cables.



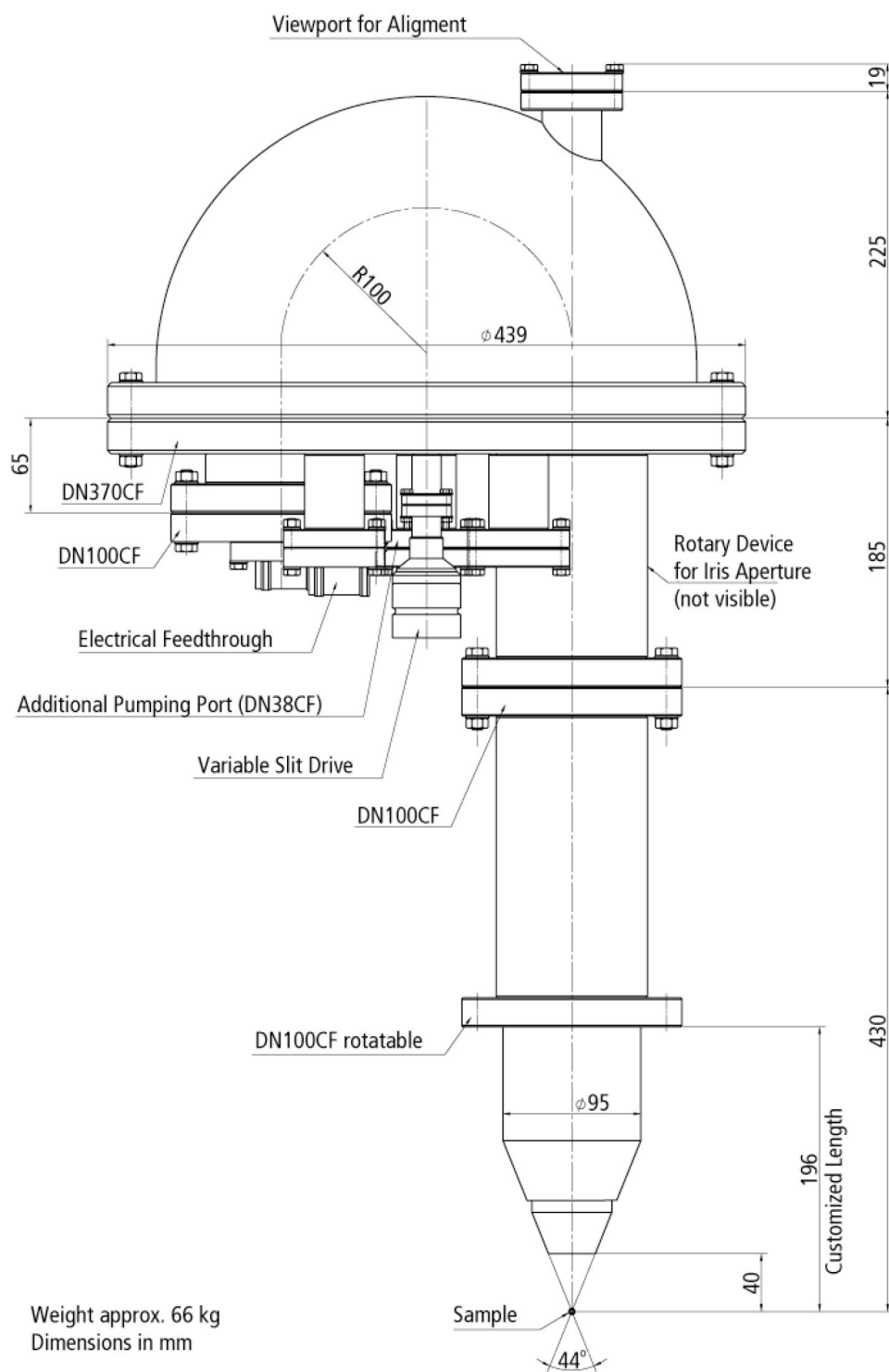


Figure 3: Analyzer Housing (PHOIBOS100)

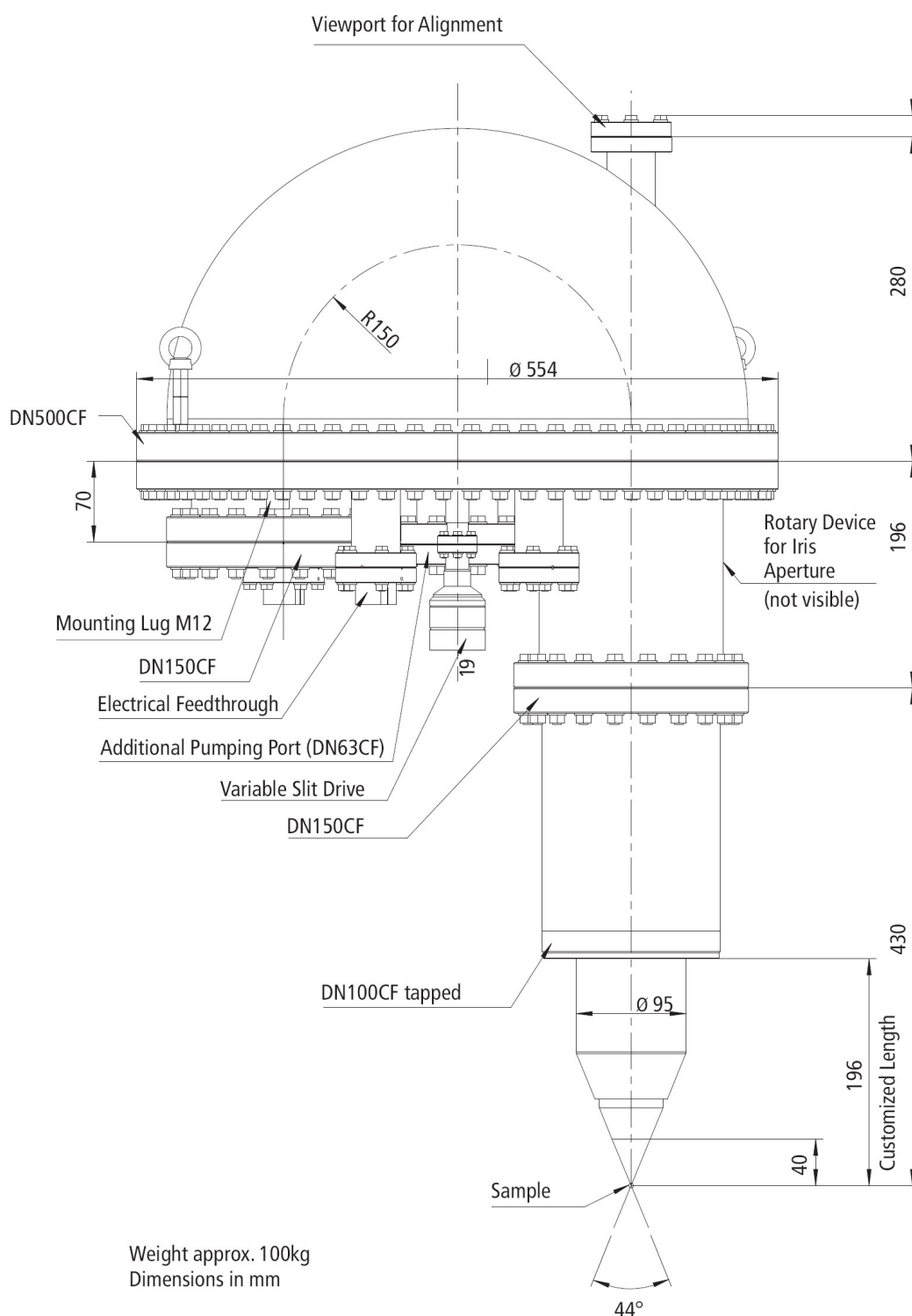


Figure 4: Analyzer Housing (PHOIBOS150)

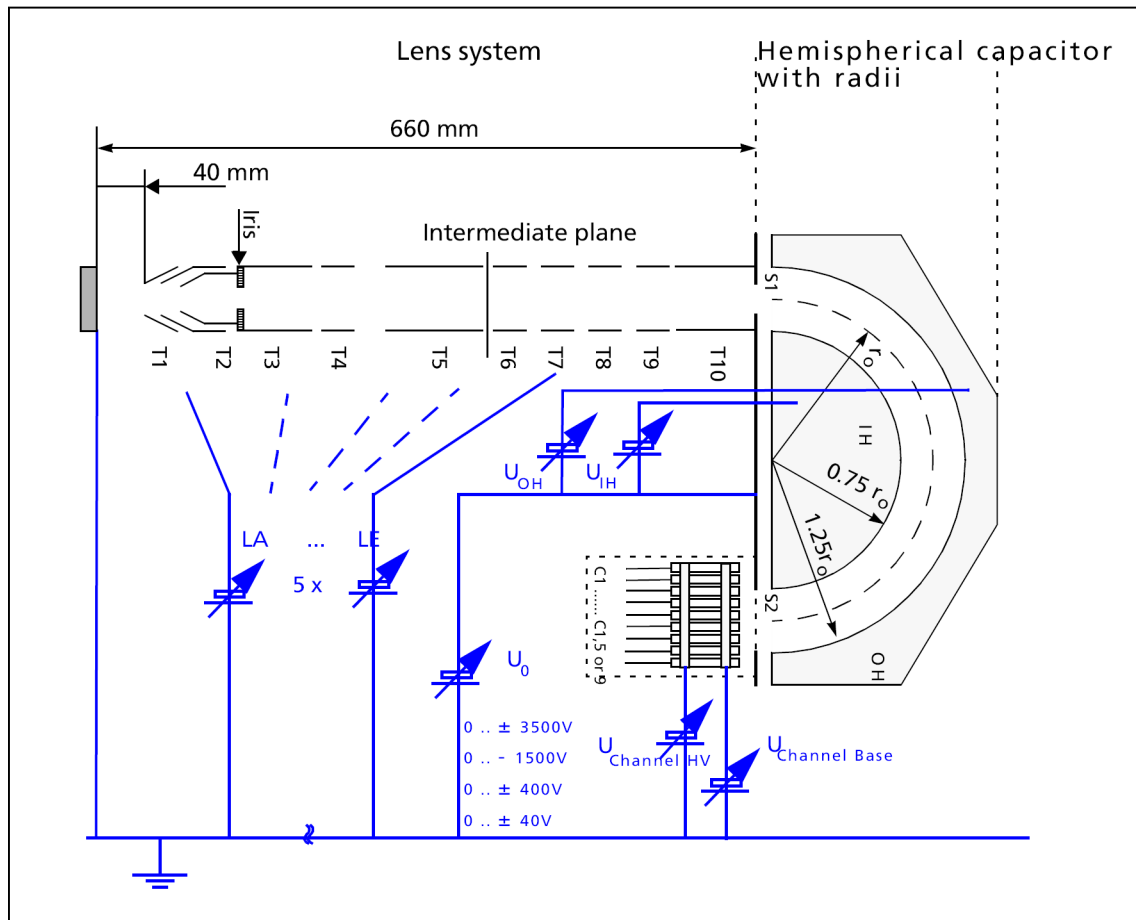


Figure 5: Analyzer Main Components and Voltage Principle

U ₀	main retardation voltage numerically equal to - kinetic energy (E _{kin}) + pass energy (E _p) + workfunction (WF)
U _{Channel HV} / Base	anode / cathode potential for the channeltrons
U _{HV} - U _{Base}	detector voltage
U _{Channel Base}	- U ₀ conversion voltage
LA..LE	lens potentials
I _H , O _H	inner / outer hemisphere
T1 to T10:	electrodes of the multi mode transfer lens
S1:	hemispherical capacitor entrance slit
S2:	hemispherical capacitor exit plane
I _H :	inner hemisphere
O _H :	outer hemisphere
r _o :	nominal capacitor radius (100 or 150 mm)
C1 to C9:	discrete collection, single / multichannel detector (1, 5 or 9 channels)
	single channel detection (SCD), multi channel detection (MCD)

Chapter

3

Spectrometer

The PHOIBOS spectrometer consists of a vacuum housing and four major internal components, which are shown in figures 3 - 5. All the parts must be contained within an Ultra High Vacuum (UHV) environment, as particles emitted from the sample surface can collide with the gas molecules changing their energy and momentum.

The internal components are:

- input lens system for receiving charged particles;
- 180° hemispherical analyzer (HSA) with 100 / 150 mm nominal radius for performing spectroscopic energy measurements;
- detector assembly for single particle detection;
- Slit Orbit mechanism with an external rotary feedthrough.
- Iris aperture with an external rotary feedthrough

The excitation source is dependent upon the technique to be used, but is commonly x-rays or other photons, electrons or ions. Before the particles pass into the hemispherical analyzer, they first pass through an electron lens system and a slit. Both the electron lens system and the slits sizes (entrance and exit) have an effect on the energy spread detected by the analyzer.

The input lens system (Figure 5: Analyzer Main Components and Voltage Principle page 10) includes ten lens tubes. For undisturbed image quality, the input lens system is grid-free. The lens stages define the analysis area and angular acceptance by imaging the emitted particles onto the entrance slit of the analyzer. Particles passing through the lenses are focused onto the input slit S1 and are retarded in the lens for subsequent energy analysis. In addition the lens stages define the acceptance angles and area for a given magnification entrance slit (see table 2 on page 16).

The lens system may be operated in several different modes for angular and spatially resolved studies to adapt the analyzer to different tasks. All lens modes can be set electronically.

For small spot analysis a lateral resolution down to 100 μm is available using the High Magnification Mode and the special Iris aperture. In the various Magnification Modes,

angle-resolving is accomplished with an Iris aperture in the diffraction plane of the lens system. using the Iris the angular resolution can be continuously adjusted while keeping the acceptance area on the sample constant.

The transmission modes are optimized for high transmission at different spot sizes of the source. The lateral resolution is worsened and the acceptance area normally not defined by the lens.

In the angular dispersive modes, the emission angle distribution is imaged instead of the real image. All lateral resolution is lost, but instead the emission angle information is easily available. These modes are intended for UPS band mapping, Fermi surface mapping or similar experiments.

In the hemispherical capacitor, the particles passing through the entrance slit S1 are focused onto the capacitor output plane S2. The radial position of the slit image in plane S2 depends on the kinetic energy of the particles in the capacitor. Particles on the central trajectory possess the nominal pass energy. They are focused to the central radial position at the exit plane S2. Particles with higher kinetic energy are focused further outside, and particles with lower energy are focused further inside in plane S2. This offers the possibility of multichannel detection, with simultaneous recording of an energy band around the nominal pass energy. Particles passing through the capacitor output plane S2 are accelerated onto the detector system C. With the multichannel detector, each channel is connected to a separate preamplifier mounted outside the vacuum. The preamplifiers are read out by the Multi Channel Detector (MCD) counter interface of the SPECS data acquisition software.

3.1 The Lens System

The multi-element, two-stage transfer lens was designed to yield ultimate transmission and well-defined optical properties. It may be operated in several different modes for angular and spatially resolved studies to adapt the analyzer to different tasks. All lens modes can be set electronically.

A lens system with the variable Slit Orbit mechanism is necessary to

- image the sample plane on the HSA entrance plane
- define the analyzed sample area and the accepted solid angle on a sample,
- accelerate / decelerate the particles with the observed energy to the pass energy.

The standard working distance of 40 mm and the 44° conical shape of the front part of the lens provide optimum access to the sample for all kinds of excitation sources. At the lens stage, the particles emerging from the sample S are imaged onto the entrance slit S1, with the sample being in the focal plane of the lens system, i.e. 40 mm in front of the first lens electrode T1. Inside the lens stage the particles pass through an intermediate image before they are focused onto the input slit S1 of the hemispherical capacitor (figure 5). At S1 the particles have been retarded by the energy difference between the nominal particle kinetic energy E_{kin} and the nominal pass energy E_{pass} .

If S1 has the dimension D1, then by theory the imaged area of the sample has the dimension DS with

$$DS = D1 / M \quad (1)$$

The magnification of the lens stage is selectable (see table 2 on page 16). The magnification is changed electrically by connecting appropriate voltages to the lens electrodes. The voltages are a function of the spectrometer voltage U_0 which is nominally equal to $E_{kin} - E_{pass} + \text{workfunction}$. E_{kin} is negative for electrons and positive for ions.

The PHOIBOS system can operate in a **Fixed Retarding Ratio (FRR)** or in a **Fixed Analyzer Transmission (FAT)** mode. In FAT mode the applied voltage to the hemispheres is defined by equation 3 on page 18. In the FRR mode, the pass energy is given by $E_{pass} = E_{kin} / R$, with the retardation ratio R.

The actual size of the analyzer sampling area DS is, in principle, given by equation 1. However, due to spherical aberration of the input lens, the image in the plane of the entrance slit is diffused. The degree of diffusion increases for a fixed magnification with the input lens acceptance angle. This means that the observed area in the focal planes of the input lens system is smeared out with increasing angle, resulting in larger sampling dimensions than given by equation 1. Thus the lens acceptance angle is selectable by the magnification modes, keeping the spherical aberration at a well known, acceptable value.

A second reason for confining lens acceptance angles are angle-resolved measurements, e.g. in tilt experiments or angle resolved photoemission investigations. Confining the lens acceptance angle is also essential in ISS, as the kinetic energy in ISS depends on the scattering angle, and thus peak broadening or double peaks appear when the lens acceptance angles are too large.

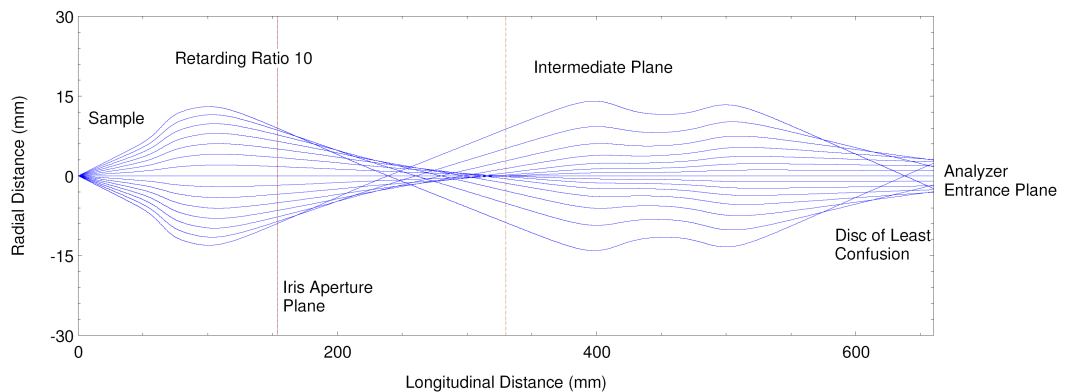


Figure 6: High Point Transmission Mode

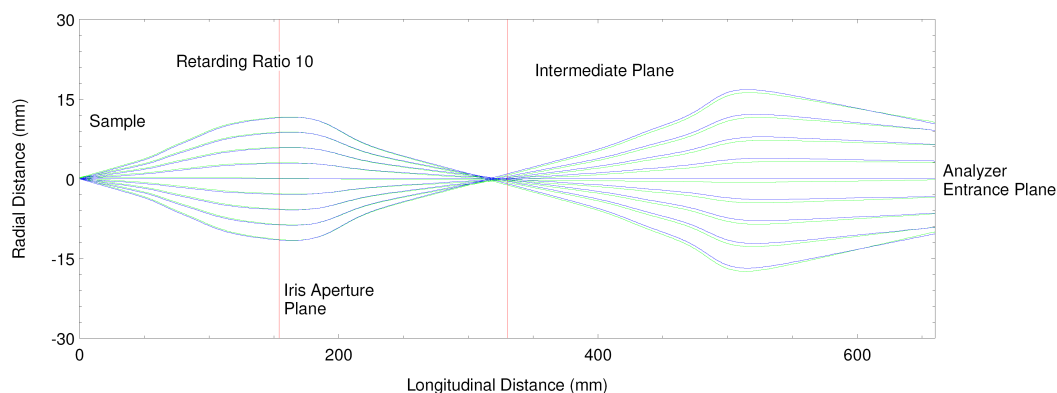


Figure 7: Medium Area Mode

High Magnification is (see Figure 8: High Magnification Mode page 15) particularly suitable for spatially resolved studies. The image plane of the sample is in coincidence with the entrance plane of the analyzer. The user can define the acceptance area of the analyzer with the entrance slit. As the trajectories of electrons emitted from the sample are influenced by electrical fields around the sample, T1 has a fixed potential, which is set to ground, after switching on the power supply. Due to lens aberrations, rays entering the lens far away of the lens axis at larger angles could find a path to the analyzer entrance. With an Iris aperture these “bad rays” can be eliminated. Furthermore the Iris Aperture can be used to continuously adjust the angular acceptance of the analyzer. For small spot analysis a lateral resolution down to 100 μm is available using the High Magnification Mode and the Iris aperture.

The magnification modes were optimized to allow very large acceptance angles for high transmission from point sources (Point Transmission Modes). In these modes angular resolution is accomplished with the Iris aperture in the diffraction plane of the lens system. Using this Iris, the angular resolution can be continuously adjusted between $\pm 1^\circ$ and $\pm 9^\circ$ while keeping the acceptance area on the sample constant. The rays close to the lens axis (paraxial rays) are focused at the Gaussian focus. Rays entering the lens at a larger angle are converged more strongly. The disc of minimum confusion is where the envelope of emergent rays has its smallest diameter. Slight under-focusing of the lens displaces the disk of least confusion to the image plane. Thereby a higher angular acceptance is achieved but the spatial resolution is worsened. This makes the Point Transmission Modes most suitable for AES, ISS and synchrotron studies. As with the High Magnification Modes, the Iris Aperture can be used to continuously adjust the angular acceptance.

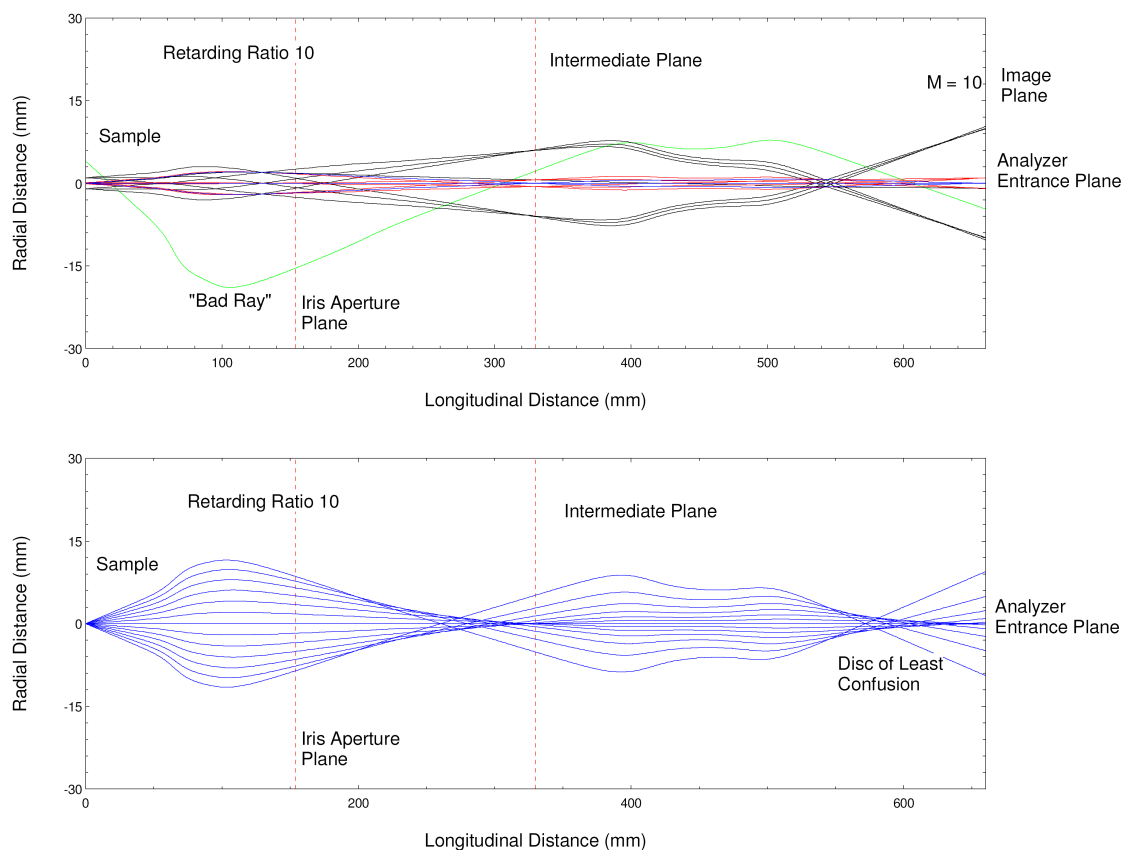


Figure 8: High Magnification Mode

Acceptance Angle	Iris Diameter
$\pm 1^\circ$	3.5mm
$\pm 2^\circ$	7mm
$\pm 3^\circ$	10mm
$\pm 4^\circ$	13mm
$\pm 5^\circ$	15.5mm
$\pm 6^\circ$	17.5mm

Table 1: Acceptance Angle vs. Iris Diameter for a Point Source

In the novel, angle-resolved Medium Area mode, electrons leaving the sample within a given angular range are focused onto the same location of the analyzer entrance independent of their position on the sample. The angular modes allow the user to optimize the angular resolution down to $\pm 0.05^\circ$ with the Slit-Orbit mechanism. With a 2-D detection system high angular resolution can be achieved in the dispersion direction of

the analyzer without restricting the acceptance angle; this mode is the ideal choice for angular dependent studies.

Magnification and angular aperture are selectable with PHOIBOS. There are many different combinations available. The lens settings can be combined with the different possible slit combinations, resulting in "lens settings" x "number of slits" possible combinations. The analyzer sampling areas and input lens acceptance angles for these combinations is given in table 2. The table shows the standard lens modes of operations. Additional acceleration modes for low kinetic energy applications are also available (HighMagnification2 and SmallArea2).

Mode	Acceptance area	Acceptance angle	Typical Application
Spatially resolved <i>High Magnification</i> <i>Medium Magnification</i> <i>Low Magnification</i>	Entrance slit size divided by M independent of analyzer settings. Slit sizes 7×20, 3×20, 1×20, 0.5×20, 0.2×20, Ø7, 3 and 1 mm Magnification M = 10 Magnification M = 5 Magnification M = 2	With Iris aperture continuously adjustable from maximum to ±1° Up to ±9° Up to ±6° Up to ±3°	Small area XPS, UPS standard ARXPS and ARUPS
Transmission optimized <i>Large Area</i> <i>Medium Area</i> <i>Small Area</i>	Optimized for different spot sizes of the source. Typical spot size Ø 5 mm Ø 2mm Ø 0.1mm	Up to ±5° Up to ±7° Up to ±9°	Large Area XPS Monochromated XPS AES, ISS and synchrotron studies
Angular resolved <i>High Angular Dispersion</i> <i>Medium Angular Dispersion</i> <i>Low Angular Dispersion</i> <i>Wide Angle Mode</i>	Slightly decreasing with increasing retarding ratio and independent of slit sizes	Entrance slit size divided by D independent of analyzer settings. Slit sizes 7×20, 3×20, 1×20, 0.5×20, 0.2×20, Ø 7, 3 and 1 mm D = 3.2 mm/° (±3° acceptance) D = 2.2 mm/° (±4° acceptance) D = 1.2 mm/° (±7° acceptance) D = 0.5 mm/° (±13° acceptance)	Low kinetic energy applications. ARXPS / ARUPS with a 2D detection system (Angular Mapping)

Table 2: Overview of the Lens Modes

For small spot analysis the iris aperture may be used to sharpen up the analysis area. The optimum settings for the iris aperture depends on the slit size and the desired quality of the analysis area. The intensity-position function for the analyzer is a Gaussian-like function, but with higher intensities in the tail regions (see figure 9). Using the iris aperture these intensities can be suppressed. The analysis area was defined to include typically between 90 - 99% of the total photoelectron signal.

Slit Size	Iris Diameter
7 x 20 mm	30 – 40 mm
Ø 7	20 – 30 mm
Ø 3	10 – 20 mm
Ø 1	2.5 – 10 mm

Table 3: Recommended Iris Values for Spatially Resolved Measurements

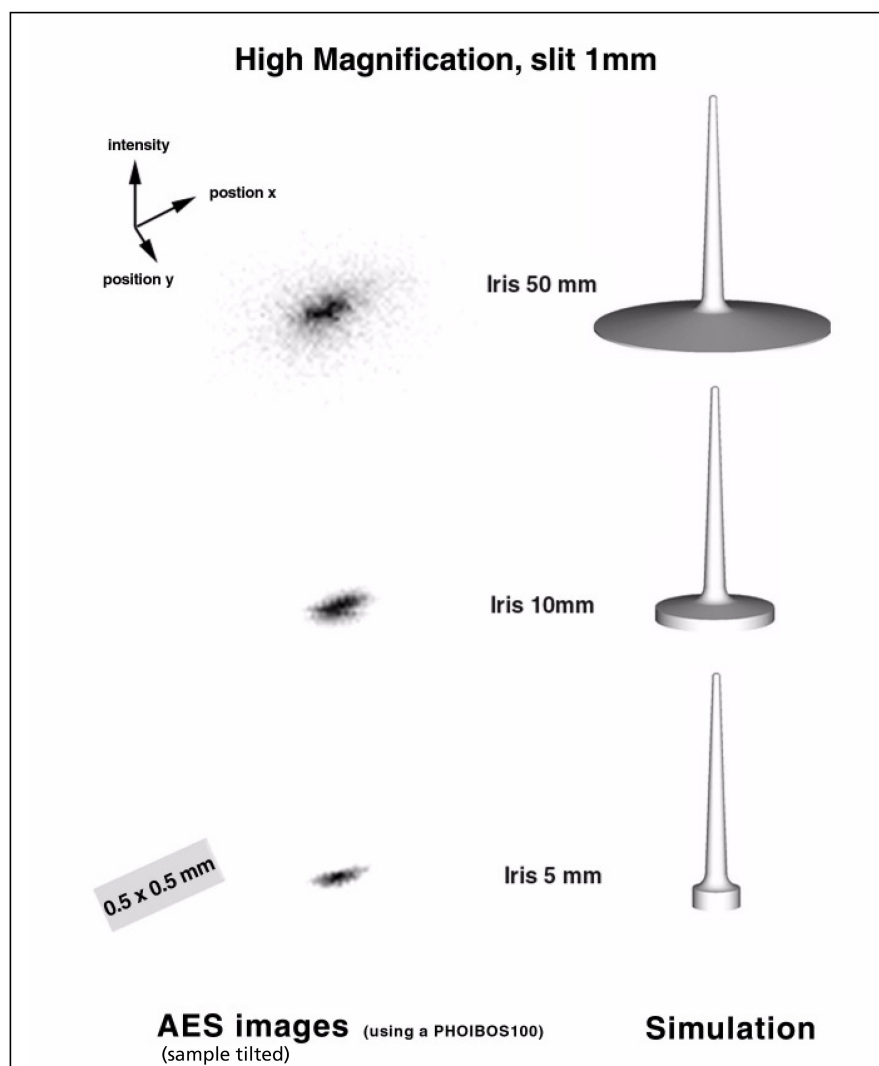


Figure 9: Typical Intensity-Position Profile with Iris Aperture

The low tail intensity forms a disc. Its integrated intensity can achieve the same order of magnitude as the intensity in the peak. Using the iris aperture these intensities can be

suppressed.

3.2 Hemispherical Analyzer (HSA)

The PHOIBOS Hemispherical Analyzer (HSA) with a mean radius R_0 (100mm/150mm) measures the energy of charged particles. Charged particles entering the HSA through the entrance slit S_1 are deflected into elliptical trajectories by the radial electrical field between the inner hemisphere R_{IN} and the outer hemisphere R_{OUT} . The radii of the PHOIBOS hemispheres are $1.25 R_0$ and $0.75 R_0$, respectively. The entrance slit S_1 and exit plane S_2 , are centered on the mean radius R_0 :

$$R_0 = \frac{R_{inner} + R_{out}}{2} = 150\text{mm} \quad (2)$$

For a fixed electrical field gradient, only particles with kinetic energies in a certain energy interval are able to pass through the full deflection angle from the entrance slit S_1 to the exit plane S_2 . Particles with higher kinetic energy approach the outer hemisphere, whereas particles with lower kinetic energy are deflected toward the inner hemisphere. Those particles, which enter the HSA normal to S_1 and move through the hemispheres on the central circular trajectory, have the nominal pass energy E_{pass} :

$$E_{pass} = (-q) k \nabla V \quad (3)$$

where q is the charge of the particle, ΔV is the potential difference $V_{out} - V_{in}$ applied to the hemispheres, k is the calibration constant,

$$k = \frac{R_{inner} R_{out}}{2R_0(R_{out} - R_{inner})} = 0.9375 \quad (4)$$

These particles reach S_2 at the nominal radial position R_0 . If the HSA accepts the half angle α in the dispersion direction, the HSA resolution or FWHM (full width at half maximum) of the transmitted line ΔE_{an} is given by

$$\frac{\Delta E_{an}}{E_{pass}} = \frac{S}{2R_0} + \frac{\alpha^2}{4} \quad (5)$$

where $S = (S_1 + S_2)/2$. This value is a constant of the analyzer.

There are additional contributions to the line width observed in the spectrum. For photo-emission lines, the main additional contributions are:

- inherent line width of the atomic level ΔE_{level} (e.g. O 1s, C 1s),

- natural line width of the characteristic radiation used for excitation ΔE_{photo} (e.g. Mg, K α , Al K α).

The observed total FWHM_{total} is given by the convolution of the single FWHMs, e.g for gaussian line widths.

$$FWHM_{\text{total}} = (\Delta E_{\text{an}}^2 + \Delta E_{\text{level}}^2 + \Delta E_{\text{photo}}^2)^{1/2} = \Delta E \quad (6)$$

FWHM_{total} is usually specified using a sputter-cleaned silver sample and recording the Ag 3d_{5/2} level, after linear background subtraction. For Mg K α excitation, the resolution at low HSA pass energies for the Ag 3d_{5/2} level is found to be

$$HM_{\text{Mg K } \alpha} = 0.8 \text{ eV} \quad (7)$$

In most practical work, a resolution of 0.9 eV is usually sufficient for high resolution investigations. For higher instrumental resolution, it is possible to use monochromatized X-radiation for excitation, e.g. mainly monochromatized Al K α radiation. For monochromatized Al K α radiation and for the Ag 3d_{5/2} level, the extreme resolution is found to be

$$FWHM_{\text{extreme}} = 0.44 \text{ eV} \quad (8)$$

To obtain the extreme resolution of 0.44 eV, the FWHM of the X-ray has to be heavily restricted, by utilizing only a small part of the X-ray monochromator spot area (due to the energy dispersion across the spot area), at the expense of a strong loss in intensity. In practice, a resolution of 0.65 eV is usually sufficient for high resolution investigations with monochromatized Al K α excitation. For monochromatic radiation, FWHM_{total} is sometimes specified recording the Si 2p_{3/2} level instead of the Ag 3d_{5/2} level, which results in smaller values of FWHM_{extreme}. due to the narrower inherent line width of the Si 2p level.

The integral signal intensity I of the measured particles (the area under the peak with a background subtracted) is proportional to product of the accepted solid angle Ω_s , the accepted sample area A_s and the HSA resolution ΔE_{an} :

$$I \sim \Delta E_{\text{an}} \Omega_s A_s = \Delta E_{\text{an}} \Omega_0 A_0 \frac{E_{\text{pass}}}{E_{\text{kin}}} \sim \frac{E_{\text{pass}}^2}{E_{\text{kin}}} \quad (9)$$

where Ω_0 and A_0 are the values of the acceptances for the HSA. They are analyzer constants. The equation results from Liouville's theorem¹.

The analyzer can be operated in two different modes:

- a) Fixed Retarding Ratio (FRR), the retardation ratio R is defined as

1 For more information there are some excellent publications on analyzers. We recommend two of them:
K. D. Sevier, Low Energy Electron Spectrometry, Wiley-Interscience, 1972
D. Roy and D. Tremblay, Design of Electron Spectrometers, Rep. Prog. Phys. 53, 1621-1674, 1990

$$R = \frac{E_{kin}}{E_{pass}} \quad (10)$$

In this mode all particles are decelerated with this same fixed factor. Therefore the pass energy is proportional to the kinetic energy. The intensity increases with kinetic energy:

$$I \sim E_{kin} \quad (11)$$

while the energy resolution decreases.

- b) Fixed Analyzer Transmission (FAT), E_{pass} and ΔE_{an} in equation 5 are adjustable constants. The signals of all particles, independent of the kinetic energy, are measured with the same resolution and the intensity decreases with the kinetic energy:

$$I \sim \frac{1}{E_{kin}} \quad (12)$$

The two modes are generally possible for all kinds of measurements. There are some applications where one of them is traditionally preferred. The FRR mode is mostly used in AES, ISS and is convenient for the measurement of a survey spectrum. The FAT mode is mainly used in XPS and UPS when detailed information is needed and the resolution should not be dependent on the energy. If E_{kin} is kept constant and the same peak is measured with different pass energies, it follows that

$$I \sim E_{pass}^2 \quad (13)$$

3.3 Magnetic Shielding

Because charged particles are influenced by stray magnetic fields (including the earth's magnetic field), it is essential to cancel these fields within the enclosed volume of the analyzer. The analyzer, the lens system and the detector are surrounded by one layer of 1.5 mm thick μ -metal to screen the external magnetic fields down to an uncritical level. The shielding factor for the analyzer region is about 35. For ultimate performance the analyzer and the lens system are constructed entirely from non-magnetic materials inside the μ -metal shielding. (see also section 4.4 - Alignment on page 40)

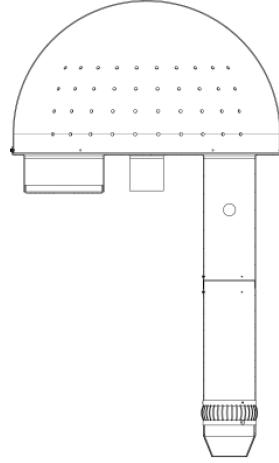


Figure 10: PHOIBOS μ -Metal Shielding

3.3.1 Magnetic Coupling for μ -Metal Chambers

A proper shielding of the front of the lens and the experiment chamber is also necessary, and to facilitate magnetic coupling to these parts, the PHOIBOS analyzer is delivered with a collar attached to the front of the lens shielding. The chamber shielding needs good physical connection to the analyzer shielding, especially for slowly charged particles. The insertion depth of the analyzer shielding has to be modified if the shielding is too far inside the chamber (normally by a non-magnetic lens protection cap added to the lens shielding). Please contact SPECS for advice regarding the μ -metal shielding.

3.3.2 Trim Coil

The PHOIBOS analyzer can be equipped with a trim coil around the outer hemisphere of the analyzer. With a coil current $|I_{\text{coil}}| < 150$ mA the residual magnetic field component within the analyzer along the lens axis can be compensated. The magnitude and the sign of the coil current depends on local terms.

The radius of gyration G for an electron with kinetic energy E_{kin} in a magnetic field B is

$$RG [m] = 3.37 \cdot \frac{\sqrt{E_{\text{kin}} [eV]}}{B [\mu T]} \quad (14)$$

At the center of a very short coil with coil current I , radius R and n windings the magnetic field is

$$B = \mu_0 \cdot \frac{n \cdot I}{2R} \quad (15)$$

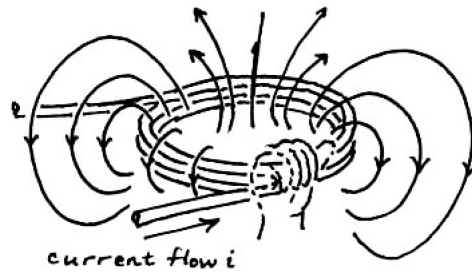


Figure 11: Magnetic Field of the Trim Coil

For the PHOIBOS 150 analyzer $B[\mu\text{T}] = 0.017 \times I[\text{mA}]$ and for the PHOIBOS 100 analyzer $B[\mu\text{T}] = 0.026 \times I[\text{mA}]$. In the case of an earth magnetic field of about $50 \mu\text{T}$ with the lens axis nearly parallel to the field one needs about 85 mA or -85 mA to compensate the residual magnetic field within the hemispheres.

To find the optimal value for the current, the signal measured by the analyzer at low pass energies ($< 10 \text{ eV}$) should be maximized by changing the coil current. If the analyzer is equipped with a coil (short-circuit of PIN 12 to ground of the analyzer feed-through) and the power supply HSA 3500 is equipped with a current module, the control software allows the setting of the coil current. In all programs the coil current setting can be found in the analyzer settings menu as an additional field.

3.4 Slit Orbit Mechanism

The slit orbit mechanism is used to manually change the entrance and exit apertures of the analyzer. The mechanism positions an entrance and exit opening of the appropriate size in the analyzer entrance and exit plane. The entrance beam is defined by a pair of slits which are about 6 mm apart. The posterior slit lies in the entrance plane and defines the analyzer energy resolution (see equation 5 on page 18), while the anterior slit serves to match the angular spread for the analyzer. For a given energy resolution and a given tolerated analysis area and acceptance angle, the largest possible slit

should be selected. This enables the highest possible count rate for these parameters and thereby a short measurement time and a good signal-to-noise ratio.

The entrance ring can be positioned directly by rotating the dial (see figure 13, "Entrance and exit slit rings (slit combination 4-B)," page 24). The exit ring on the other hand is positioned indirectly by the use of the entrance ring (see Figure 13: Entrance and Exit Slit Rings (Slit Combination 4-B) page 24). Through this arrangement one can choose the entrance and exit slits independently using the same rotary drive.

The PHOIBOS analyzers, beginning with release 5, have 8 entrance and either 2 exit slits in the standard configuration or 3 upon request. The entrance slit positions are indicated by numbers (1-8) on the external rotary dial (see Figure 12: External Rotary Dial for Positioning page 24). The exit slit positions are indicated by letters (A-B or A, B and C). These indicators correspond to those which appear in the region settings of the analyzer control software SpecsLab 2. Whenever the entrance and exit aperture is changed the positions must be entered in the region edit dialog. The arrangement of the slits may be other than those shown in table 4 on page 23. The actual arrangement present in an analyzer appears in the slit selection dialog of the control software delivered with the analyzer.

Entrance S1			Exit E2	
Slit Number	Slit Size		Slit Number	Slit Size
1	0.2 x 20 mm	Free choice of each slit combination except combination 8-A and 1-C	A	0.25 x 20 mm
2	0.5 x 20 mm		B	Open (for CEM each channel is 7x21 mm)
3	1 x 20 mm			
4	3 x 20 mm		C	3 x 20 mm
5	7 x 20 mm			
6	dia. 1 mm			
7	dia. 3 mm			
8	dia. 7 mm			

Table 4: Standard Slit Configuration

(May be different for your analyzer, please check the slit selection dialog of the SpecsLab2 software.)



Figure 12: External Rotary Dial for Positioning

The indicators on the rotary dial are used for positioning, but it must be taken into account that the rotary dial has some backlash. The correct slit positions are defined by spring loaded indexing balls. Because of the rotary feedthroughs backlash, rotate it beyond the desired position until the indexing ball snaps in.

After positioning the dial, jog it back and forth to ensure that the index is probably engaged. The correct positioning of the entrance slits can also be checked by looking through the view port. In a configuration with two exit slits, the positioning of the exit slit is not critical because of the end stops. In configurations with a third exit opening please be aware that positioning of slit B can be tricky and require some practice. An analyzer configured with two exit slits can be reconfigured on request.

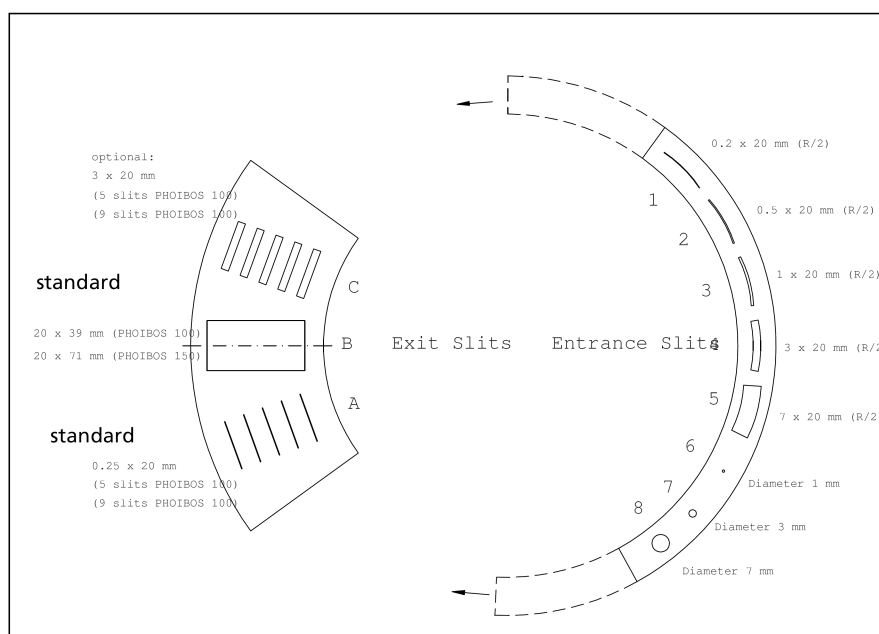


Figure 13: Entrance and Exit Slit Rings (Slit Combination 4-B)

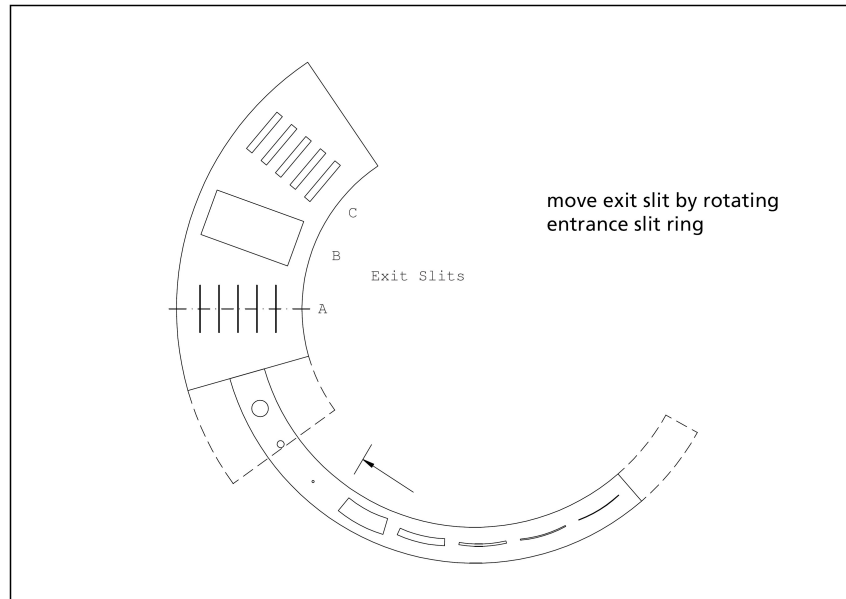


Figure 14: Exit Slit Selection

3.5 Single and Multichannel Detector (SCD) / (MCD)

The detector consists of the following parts:

- an arrangement of Channel Electron Multipliers (CEMs), 1 for SCD, 5 or 9 for MCD, consisting of discrete collectors, specially screened against external HF-signals for maximum noise rejection;
- a multi-pin ceramic, high voltage vacuum feedthrough, specially designed for low cross talk;
- SCD / MCD preamplifier;

3.5.1 Principles of Detection

Due to the spherical symmetry of the HSA, a one-to-one image of the circularly shaped entrance slit with a radius of curvature R_0 exists in the exit plane for monochromatic electrons with a nominal pass energy E_{pass} . The images of electrons possessing different energies within the HSA are concentric circles. In a first order approximation, the radial image position R for electrons with kinetic energy E_k is given by

$$\frac{R - R_0}{R_0} = \frac{E_k - E_{pass}}{E_{pass}} \cdot \frac{D}{R_0} \quad (16)$$

where D is the has dispersion. The theoretical value for D is

$$D = 2 \cdot R_0 \quad (17)$$

The experimentally determined dispersion value can be slightly different, mainly due to fringing fields at the edges of the analyzer.

Multichannel detection is performed by appropriately arranging 5 or 9 CEM's as collectors with 5 or 9 exit slits on concentric circles in the exit plane. The radial distance between neighbouring exit slits ΔR is selected to meet the requirement of a constant kinetic energy difference between neighbouring channels ΔE_k . The number of particles N_n arriving at each collector C_n is counted separately, and these numbers are stored and preprocessed in the data acquisition unit.

By sweeping the spectrometer voltage U_0 , the electron path is moved across each collector channel step by step, and in this way each collector records a complete spectrum, with a fixed energy offset between neighbouring channels. In principle, by sweeping the spectrum once over the detector area, 5 or 9 parallel spectra are recorded simultaneously. As the kinetic energy E_n of the particles arriving at collector C_n is known from equation 16, the number of particles from each channel, belonging to the same kinetic energy can simply be added, resulting in a total number of particles for each kinetic energy.

3.5.2 Coherence of E_{pass} and Step

From the analyzer energy dispersion equation, the energy difference ΔE_k between neighbouring channels at the distance ΔR one from another is

$$\Delta E_k = \frac{\Delta R}{D} \cdot E_{pass} \quad (18)$$

or

$$E_{pass} = \frac{D}{\Delta R} \cdot \Delta E_k \quad (19)$$

where D is the analyzer dispersion.

Especially in the FRR mode, where the pass energy changes throughout the spectrum (and thus the energy difference between neighbouring channels) a calculation of the detected energy of the particle is necessary. Therefore a software routine calculates the particle number N_n in channel C_n at the nominal kinetic energy, by interpolation between the numbers actually measured in channel C_n at the measuring energies nearest below and nearest above the nominal energy.

This algorithm is unequivocal, because there is never more than one nominal energy between two measured energy positions. Due to the interpolation routine, there is no restriction on the energy step due to analyzer performance. Power supply performance (DAC steps, etc.) limit the possible step widths and ranges. The software validates the values to the nearest allowed values.

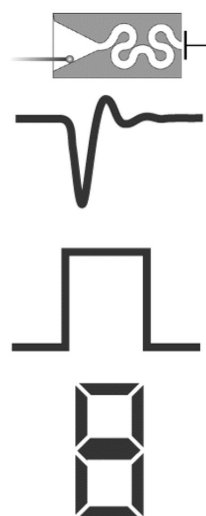
3.5.3 Electron Multiplication

A Channel Electron Multiplier (channeltron or CEM) is a high gain device for detecting energetic particles such as electrons and ions, or radiation. The CEM consists of a small, curved glass tube. The inside wall is coated with a high resistance material. The resistive material becomes a continuous dynode when a potential is applied between the ends of the tube.

The impact of a charged particle results in secondary electrons that are released from the CEM wall. These electrons are accelerated by the high voltage connected to the CEM and release additional secondary electrons by impact with the wall further along in the CEM. This effect is repeated successively, until finally an "electron cloud" is present at the exit of the CEM.

The average number of electrons leaving the CEM assembly per incident particle is called the gain G . For single particle detection, the gain has to be selected high enough to use the CEM's in "saturated" operation, i.e. each incident particle releases an electron cloud at the exit of the CEM arrangement whose charge is independent of small changes in multiplier voltage. The saturated operation is necessary for sufficient noise rejection in single particle detection. Usually the minimum gain for saturated operation is about 107, i.e. an electron cloud of more than 107 electrons leaves the CEM.

The electron cloud emitted is accelerated onto the collector electrode of the CEM, and the charge pulse carried by the electron cloud is detected as originating from one incident particle and counted in the preamplifier channel.



One or a set of CEM's, is used in a special arrangement as an electron multiplying component for the PHOIBOS analyzers. The CEM's are all mounted in parallel as a unit on a feedthrough flange. Particles passing the exit aperture are accelerated to an appropriate kinetic energy onto the CEM. The particle energy can be calculated as described in section 3.5.4, "Conversion Energy" on page 32.

The Working Point of the CEM's

The operating point for a channel electron multiplier (CEM) in the pulse counting mode is usually determined by the point at which a plateau is reached in the count rate vs. voltage characteristic. Within the plateau all electrons collected at the input of the CEM give an electron pulse at the output, high enough to be detected by the electronics.

Additional increases in voltage raise the gain, but the count rate remains essentially constant. Eventually, a point is reached where ion feedback becomes significant due to very high gain, and the count rate increases rapidly. **This will significantly reduce the lifetime of the channeltron.**

The **Default Detector Voltage** is normally given in the specification protocol of the analyzer and **should be changed carefully**. A detector scan gives the actual status of the detector unit (see Figure 15: Detector Sweep, Count rate vs. Voltage on page 29). Note:

Please choose a moderate end value of the detector voltage scan to prevent rapid aging of the detector!

The optimum operating point is about 50 - 100 V beyond the plateau of the curve (figure 15, page 29). As the multiplier ages, the knee moves to the right and the voltage must be increased. In general, CEM lifetime (see figure 16, page 30) depends on the number of counted electrons, thus it will vary as a function of specific application and environment, but is typically on the order of about one year at 40 hours operation per week.

For multi channel detection systems each channel should be displayed. The start value for the detector voltage for counting depends on total number of accumulated counts (see section 3.5.3.1 "Extended CEM" on page 29) and the threshold level of the preamplifier. The sensitivity of the preamplifier channels can be varied using a discriminator threshold (the value is factory preset, recommended discriminator voltage see section 9.1.1 "Discriminator" on page 79). Large differences between the channels can be equalized by setting the threshold for the channels individually. These differences may be caused by the varying sensitivities of the CEM's. **Pay attention to the detector voltage value in the Specification Report sent with the analyzer.**

The detector check should be done monthly (choose kinetic energy of 400eV).

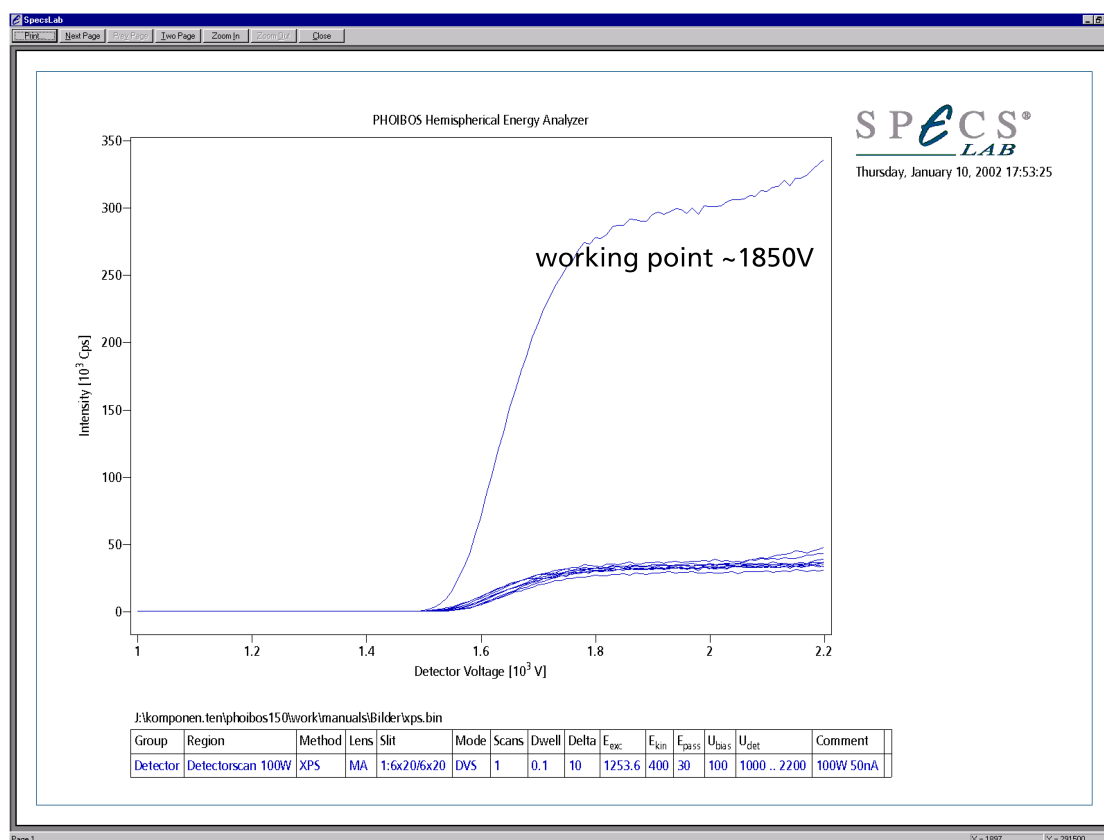


Figure 15: Detector Sweep, Count rate vs. Voltage

The pulse output depends largely on the applied voltage and, in practice the gain is an increasing function of the applied voltage until the gain reaches about 10^7 : after which point increasing the voltage further will cause the eventual breakdown of the CEM. (With a properly configured oscilloscope, i.e. impedance 50 Ohm, the necessary pulse height can be checked, usually between 2-10mV, see section 9.1.2 , "Amplifier Check" on page 76.)

For the PHOIBOS analyzer, an input of one electron to the CEM produces an output pulse that contains at least 10^7 electrons and lasts for approximately 10 nanoseconds.

3.5.3.1 Extended CEM

The standard detector systems of the PHOIBOS analyzer series are based on the extended range CEM. This device is a specially formed and treated glass tube which has the effect of multiplying a single electron at the input to a pulse of around 10^8 electrons at the output. Because of the low resistance (about 50 M Ω) the extended range CEM's are suitable for extremely high count rates.

A voltage between 2.5 - 3.5 kV across the cone and the tail-end initiates the electron multiplication. Electron multiplication is produced by the emissive layer along the inner

surface of the channel. The gain is governed by the detector voltage and the condition of the emissive layer. The condition of this layer changes with usage and to compensate for a drop in emissive quality of the surface, an increased detector voltage can be applied keeping the overall gain constant. If the detector voltage has reached the limit of 3.5 kV the CEM is at the end of its life and needs replacing.

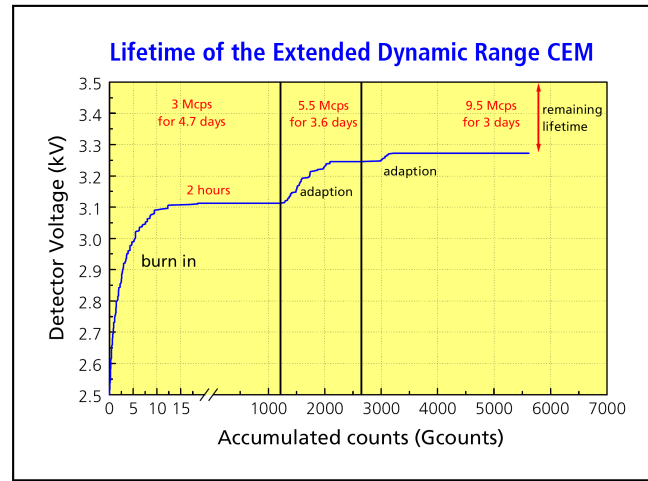


Figure 16: Lifetime of the Extended Dynamic Range CEM

- While a CEM is not counting, residual gases in the system are adsorbed onto the channel walls, which are kept clean by electron bombardment during operation.
- When initially running a new CEM it needs approximately 20×10^9 counts for conditioning. Once properly conditioned, or "burned in", the surface on the semiconducting glass channel is quite stable.
- The test results suggested that accumulations to 5×10^{12} counts and higher can be expected without serious degradation.
- The extended range CEM's are suitable for extremely high count rates without serious degradation.

3.5.3.2 Linearity of the CEM's

Inadequate design may cause electron analyzers to show non-linear behavior. For an ideal counter with a non-extended dead time τ the measured count rate N' and the true count rate N are given by

$$N' = N / (1 + N\tau) \quad (20)$$

With the PHOIBOS SCD analyzer the count rate as a function of the Auger electron beam current has been measured using both the standard and extended dynamic range CEMs. For a non-extended dead time counter the ratio N'_1 / N'_2 of two spectra N'_1 and N'_2 (measured at two different beam currents $A \times I$ and I) the spectral ratio is given by

$$N'_1 / N'_2 = A + (1 - A) \times \tau \times N'_1 \quad (21)$$

Therefore, from a spectral ratio plot the dead time can be determined. The detection efficiency N'_1 / N_1 can now be calculated

$$N'_1 / N_1 = N'_1 / N'_2 \times (1 - N'_2 \tau) / A \quad (22)$$

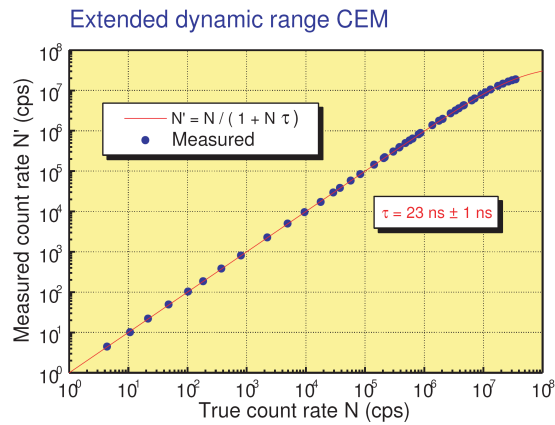


Figure 17: Linearity Plot for the new Extended Range CEM

The count rate N' was measured for different beam currents I . From the low count rate region, the conversion factor c ($N = c \times I$) can be calculated.

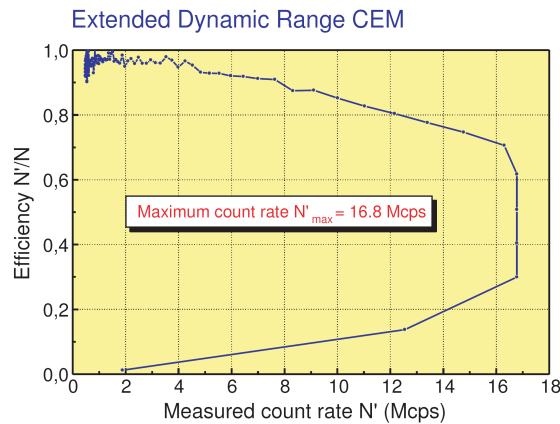


Figure 18: Efficiency Plot for the new Extended Range CEM

From a spectral ratio plot of two spectra measured at different beam currents the detector efficiency N' / N was calculated. With increasing count rate the mean gain of the pulse height distribution will decrease. For some critical value the distribution starts to fall below the discriminator threshold.

Conclusions:

- The maximum (measured) count rate detectable is 5.6 Mcps per channel for the standard CEMs and 16.8 Mcps for the extended range CEMs.
- The linearity and the non-extended dead time behavior for the standard CEMs up to 1 Mcps and for the extended range CEMs up to 10 Mcps true count rate is verified. Up to these count rates no significant deviation from linearity could be observed with the PHOIBOS detection system (CEMs and PCU 300 detection electronics).
- From pulse height distribution measurements the findings are that the mean gain from the extended range CEMs is much less sensitive to increases in the true count rate. This is the reason for the extended working range.
- The detector voltage required to operate the extended range type CEMs is 300 V higher than that for the standard CEMs.
- The high current AES survey spectrum shows the high count rate capability of the PHOIBOS detection system with the extended dynamic range CEMs.

3.5.4 Conversion Energy

The detection efficiency of a CEM with respect to a particle is defined as the probability of this particle or photon producing an output pulse. The detection efficiency curves for electrons, positive ions and UV-light in figure 19 are based on published data and allow an approximate estimation of the efficiency in the given energy and wavelength range.

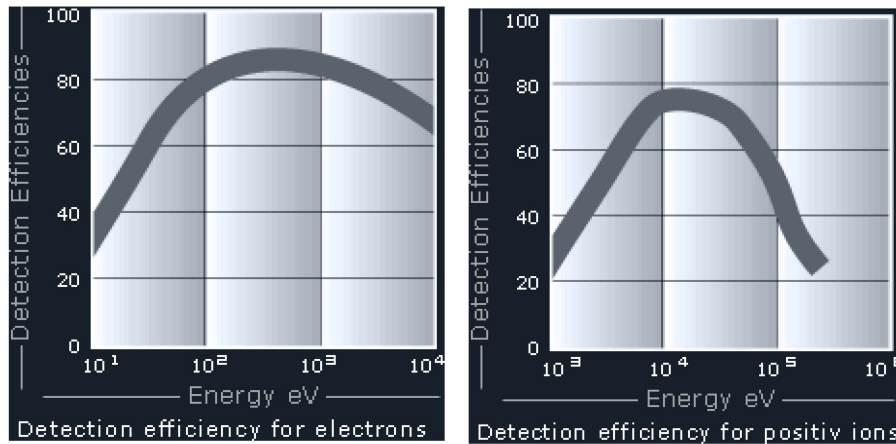


Figure 19: Detection Efficiency for Electrons and Ions

The BIAS voltage produced in the HSA3500 determines the conversion energy

$$E_{conv} = -q U_{BIAS} + E_{pass} \quad (23)$$

of the charged particles impinging on the CEM. The proper conversion voltage has two requirements which must be simultaneously fulfilled:

- the particles energy should be suitable for maximum yield of secondary electron emission at the impact on the CEM wall. For electrons this is roughly in the energy range between 100 - 800 eV.
- For ions, the yield increases with the kinetic energy roughly up to 10 keV.

Standard settings are:

- for electrons: $U_{BIAS} = +90$ V.
- for ions: $U_{BIAS} = -2000$ V.

3.5.5 Spectrometer Voltage U_0

The main retardation voltage of the spectrometer U_0 is numerically equal to the difference between kinetic energy (E_{kin}) and pass energy (E_{pass}) + Work Function (WF). Because of the different polarity of the lens and hemisphere voltages in the different analyzer modes (XPS, AES, UPS or ISS) the value U_0 is calculated by

$$U_{groundplate} = (E_{kin} - E_{pass} - WF) / q \quad (24)$$

The spectrometer voltage is applied on a groundplate and lens element 10 (Figure 5: Analyzer Main Components and Voltage Principle page 10 and Figure 33: Schematics of the 12-pin Analyzer Feedthrough page 72 as well as the comments in section , " " on page .

Example:

WF=4.5 eV, E_{kin} =1000 eV, E_p = 100V

- for electrons: $U_0 = - 895.5 \text{ V}$
- for ions: $U_0 = + 895.5 \text{ V}$

3.6 Work Function

The basic energetic properties are shown in figure 20 for the example of the measurement of photoelectrons.

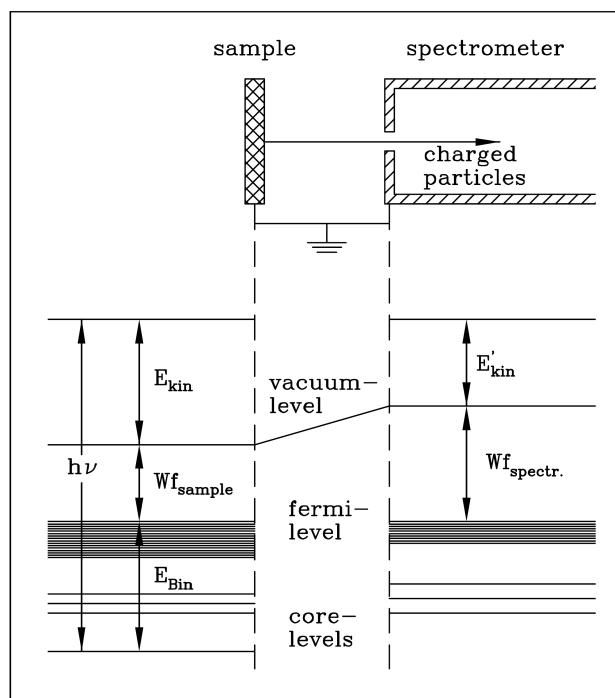


Figure 20: Energy Scheme – Photoelectron Spectroscopy

The spectrometer and the sample are connected to ensure that the Fermi-energies are at the same reference level. The binding energy of the electrons is given by

$$E_{bin} = h\nu - E_{kin} - Wf_{sample} \quad (25)$$

The energy E'_{kin} (see figure 20) is measured by the spectrometer and after calibrating the work function of the spectrometer, the binding energy of the sample relative to the Fermi level can be measured without knowing its work function because

$$E_{kin} + Wf_{sample} = E'_{kin} + Wf_{spectr} \quad (26)$$

Typical values of the analyzer work function are between 4 eV and 5 eV. The compensation is performed by addition through the software (see SpecsLab software manual). For fine adjustment, use UPS mode measurement. Please check the analyzer settings in

the SpecsLab2 program. Adjust the desired voltage ranges separately (select the voltage range in the analyzer settings before the measurement).

Please take note of section 7.4 "Work Function Calibration with UPS" on page 61.

Chapter

4

Installation**4.1 Unpacking**

All analyzers and associated electronics are carefully packed before leaving the factory. Carefully examine all packages for damage, especially the shock and tilt sensors inside and outside the transport container. If damage is suspected please contact SPECS immediately for further instructions on what to do next.

The analyzer should remain in its protective packaging until it can be bolted directly onto the system. Take great care when unpacking to prevent damage. Do not rest the analyzer on the ceramic feedthroughs, lens or the viewport. Handle parts on the vacuum side of the flange seals using normal UHV protection, i.e. wear gloves and use clean nonmagnetic tools.



The analyzer is evacuated. In order to avoid any damage, modest vacuum conditions (10^{-3} mbar) have to be maintained. Please check vacuum conditions every three months when storing the analyzer.

Please note the parts list in section 2.1 “System Description, Package Contents” on page 5.

4.2 Mounting the Detector**Caution!**

Remove the three transportation locks before mounting the detector.

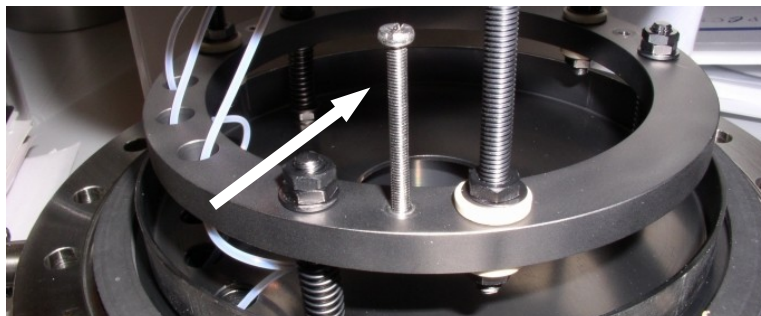


Figure 21: Removal of the Transportation Locks

Release the four M8 screws, vent the detector with N₂, and pull it carefully out of the storage housing. If one looks into the opened detector flange of the analyzer from the bottom side, you can locate the 'alignment hole' for the detector. Align the pin at the top of the detector assembly with the corresponding hole in the ground plate (see figure 21). Be sure that the detector is nearly parallel to the ground plate and push the detector carefully into its seat. While pressing the detector into its seat, make sure that the distance between the two flanges is approx. 4 mm before you feel that the springs are compressing. This will ensure that the ceramic spacers contact the ground plate. Hold the detector in its position and tighten the flanges together with the M8 screws.

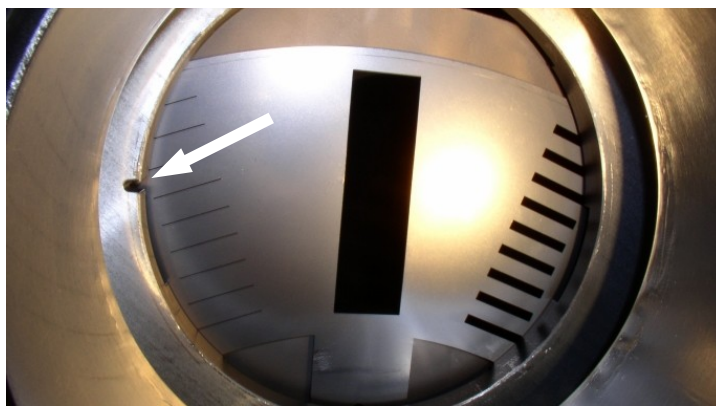


Figure 22: Corresponding Hole in the Ground Plate

4.3 Achieve the Maximum Lifetime of a Channel Electron Multiplier

To achieve the maximum lifetime of a channel electron multiplier the rules listed below should be strictly followed.

- After a bakeout, the analyzer needs 2-3 days to cool down. If channel electron multipliers are operated at higher temperatures ($> 60^{\circ}\text{C}$) they can suffer severe damage. Some channel electron multipliers will lose gain and exhibit a markedly higher detector plateau. The interior parts of the PHOIBOS will cool down significantly slower than the housing. It is recommended to wait for a complete cool down of the detector assembly; at least 2 days. Even if the analyzer housing is cold, any internal parts or insulators may still be too hot for safe operation. It is imperative that all users are aware of the issue and take the necessary precautions.
- During first use after bake out, rapid desorption of surface adsorbed gas will occur from the walls of the channel electron multipliers, so care should be taken not to use the detector at full channel electron multiplier voltage and full intensity within the first few hours of operation. We recommend to increase the detector voltage over a period of 1.5 hours up to the recommended value. Use the SpecsLab mode Detector Voltage Scan and set start, end, step and dwell time parameter for this procedure.
- During the first few days of operation of a new detector, it is recommended that high output currents are avoided (i.e. inputs above 1 Mcps). Taking this initial burn-in precaution can prevent premature failure.
- Choose a moderate value of the detector voltage to prevent rapid ageing of the detector. The optimum operating point is about 50 - 100 V beyond the plateau of the intensity versus detector voltage sweep, not more.

- Backstreaming from oil diffusion pumps or roughing pumps has to be avoided at all costs. It is strictly recommended to use cold traps and molecular sieve traps and maintain them according to manufacturers specifications.
- Channel electron multipliers can be degraded by exposure to various types of hydrocarbon gases which raises the work function of the surface and hence causing gain degradation. Operation in a clean vacuum environment of 5×10^{-8} mbar or better is a must in order to ensure the long-life characteristics of these devices.
- Other gases containing F, S and Cl, which may decompose under electron bombardment must not enter the detector area.
- Due to the hygroscopic nature of the doped lead glass, it is important that the channel electron multipliers are not exposed to air for more than one day. Dry nitrogen should be used to vent the system.
- High intensity sources like electron sources needs special care. Start with lowest source strength and narrow slit settings to avoid possible degradation.
- Measurements at the secondary electron cut-off needs special care. Use low pass energies (3- 5 eV) and small slit settings to avoid possible degradation.

4.4 Alignment

Non-shielded chambers need no special modification of the lens protection (analyzer shielding).

For shielded chambers, the shielding needs a good physical connection to the analyzer shielding, especially for slow ($<100\text{eV}$) charged particles. The insertion depth of the analyzer shielding has to be modified if the shielding is too far inside the chamber (normally by a non-magnetic lens protection cap added to the lens shielding). Please contact SPECS in this case.

For XPS, the distance between the X-ray source and the sample should be minimized to obtain maximum count rates. The distance between the sample and the lens T1 must be fixed to the **working distance (sample distance) of 40 mm** (see also section 4.4 , "Alignment" on page 40). None of these three parts should be in physical contact with one another.

The performance of the channeltron decreases with time when exposed to air. Try to minimize the time between its installation and evacuation the system.

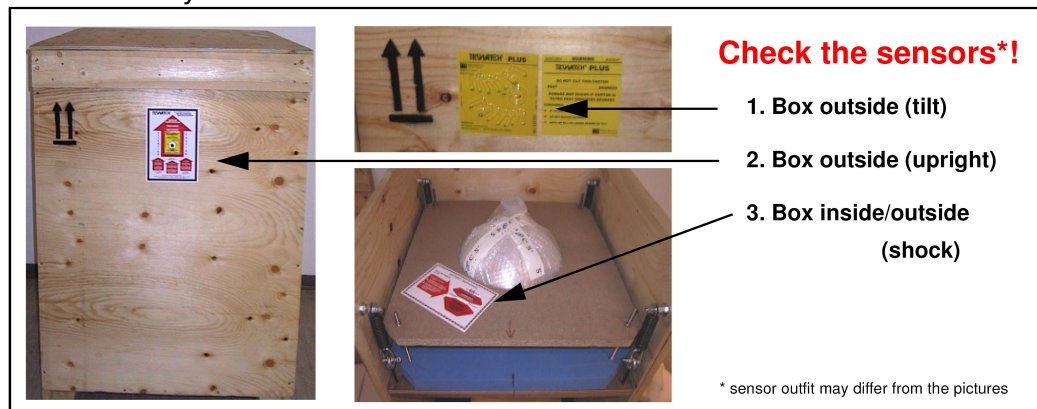
4.4.1 Analyzer Alignment

After mounting the analyzer on the chamber a check of the sample positioning via the analyzer viewport should be done. Use the iris aperture to locate the real center position of the lens system in respect to the desired acceptance area on the sample. Please contact SPECS if you see a visible offset between this two positions.

Note that the analyzer was adjusted to the mounting flange axis during specification.

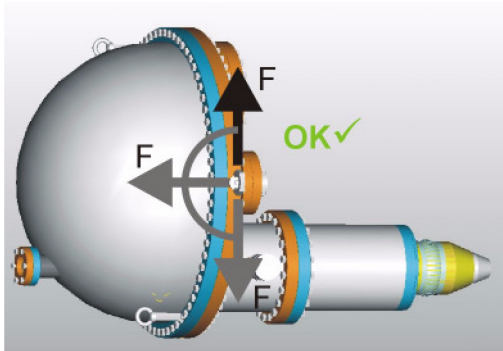
4.5 Vacuum Installation

1. Open the transport box carefully.
2. Check the shock and tilt sensors. If any sensor is discolored, please inform SPECS immediately and await further instructions.

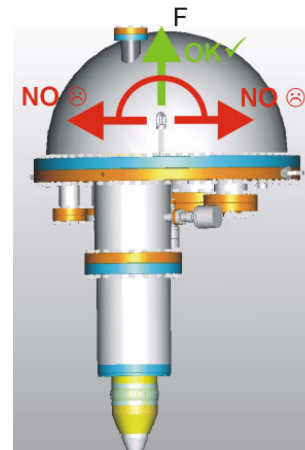


3. Remove the shipping frame.
4. Carefully lift the analyzer out of the box. Be aware of outstanding parts while pulling out the analyzer. In particular the rotary feedthroughs are sensitive to physical jostling / bumping / vibrations. Because of the analyzer weight of ~ 65 (PHOIBOS100) / 95 kg (PHOIBOS150).

Specs recommends using a hydraulic lift, as in the photo to the right.



When mounting horizontally, use the hooks as shown in the picture above.



When mounting vertically, use the hooks as shown in the picture above. Never use these for mounting horizontally!

Figure 23: Mounting Tips

5. Keep the analyzer in a stable position, if possible keep the same orientation as in the transport box (soft lie down is allowed).
6. The analyzer is evacuated. Vent via valve at the protection housing. SPECS recommends using a dry dust free venting gas like nitrogen to avoid particle or water intrusion. Remove the lens protection housing from the analyzer mounting flange. Do not touch any vacuum parts without gloves.
7. Check the working distance of the analyzer (**40mm between sample and the top of the lens system** -> insertion depth in your chamber).
8. Tighten the delivered stay bolts (PHOIBOS150) on the analyzer mounting flange and prepare the necessary mounting parts (screws for PHOIBOS100, washers and nuts).
9. Insert a new DN100CF copper gasket into the vacuum chamber flange.
10. Center the analyzer mounting flange above the vacuum chamber flange.
11. Introduce the lens system into the vacuum chamber flange very slowly. Do not use any force.
12. During the introduction check all other components in the vacuum chamber for possible physical damage. During the introduction check all other components in the vacuum chamber because of possible physical damage. All rotary feed-

- throughs are fixed with the locking screw during transport. Make sure to unlock them before using the rotary feedthroughs and lock them again afterwards.
13. Adjust the analyzer at the vacuum chamber flange (Check section 4.4 "Alignment" on page 40).
 14. Bolt the analyzer at the vacuum system flange with the delivered screws, washers and nuts.
 15. **For PHOIBOS150 and μ -metal chambers:** Do not release the lifting gear until the analyzer is supported by an additional supporting post!
 16. Check the physical stability of the supporting post and the system frame and release the lifting gear.
 17. Use the iris aperture and the analyzer view port to locate the real center position of the lens with respect to the desired acceptance area on the sample.
 18. Evacuate the chamber to a pressure of below 10^{-5} Pa (10^{-7} mbar) and bake out (see section 4.6 "Baking Out" on page 43).
 19. Check the vacuum before and after bakeout.
 20. Connect the analyzer as described in the analyzer manual. (Figure 2: Connection Scheme page 7)
 21. Before operating the analyzer wait for complete cool down (good thermal insulation inside the analyzer leads to a complete cool down time of about 2-3 days, at least 2 days cool down is recommended).

4.6 Baking Out

The vacuum chamber has to be baked out to get good UHV in a reasonable time. The temperature during the bake can be up to 200° C. A reference thermocouple for the temperature measurement should be attached to the detector flange (figure 24)!

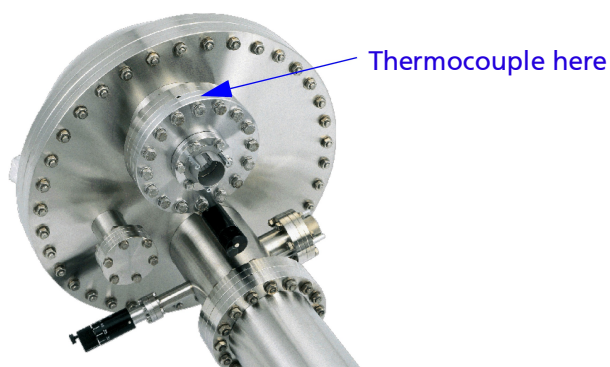


Figure 24: Detector Flange

Before baking out:

- Switch off the HSA3500 control unit.
- The analyzer box (fixed at the lens housing, see figure 4) should be removed as well as,
- the detector box (preamplifier) with connection cables (Channel HV, Channel Base, CAN)

A bakeout time between 24 hours and 48 hours (first time) is recommended.

After a bakeout the analyzer needs two days to cool down. If channeltrons are operated at higher temperatures (> 340 K) they can suffer damage. Some channeltrons will lose gain and exhibit a markedly higher detector plateau. The interior parts of the PHOIBOS will cool down significantly slower than the housing. It is recommended to wait for a complete cool down of the detector assembly; approximately 2-3 days. Even if the analyzer housing just feels warm, any internal parts seated on isolators may still be too hot for safe operation. It is imperative that all users be informed of this issue and take the necessary precautions.

The multiplier will degas at first operation after bakeout, so care should be taken not to use the detector at full multiplier voltage and full intensity within the first few hours after bakeout. We recommend increasing the detector voltage over a period of 1.5 hours for the first use and over a period of 10 min for subsequent times after bakeout. (Use the SpecsLab mode Detector Sweep and set start, end, step and dwell time parameter for this procedure.)

4.7 Electronic Units Installation

The electronic units have to be installed into a 19"-cabinet rack. Good air circulation within the cabinet must be ensured. For wiring of the electronics follow figure 2 (page 7).

Note the following:

1. Connect all units to the same power strip.
2. The power strip must be provided with a protecting device according to, and meeting all safety regulations.
3. A grounding bar (copper, brass) with a minimum cross section of 6x6 mm² has to be installed inside the cabinet. The electronic unit has to be connected to this grounding bar.
4. The grounding bar inside the cabinet has to be connected to the system (e.g. to the analyzer housing) by a ground cable of a minimum cross section of 10 mm².

The connections between analyzer, control unit and computer are described in section 2.2, "Electrical Connections" on page 6. All connections described above have to be made before the initial operation of the system.

4.8 SpecsLab Hardware and Software Installation

Read the instructions (e.g. section 2.2 , "Electrical Connections" on page 6 and SpecsLab2: Chapter 1: Installation) carefully before installing SpecsLab2 and evaluation program. You will find the manuals and other instructions in pdf-format on the installation CD as well.

Please do not hesitate to contact SPECS for more detailed information.
support@specs.de or
phone +49 (30) 467 824 - 0 or - 88)

Chapter

5

Unit Operation

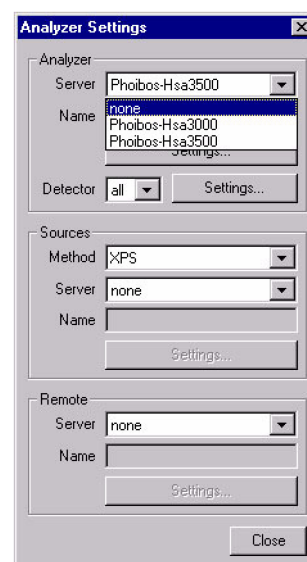
5.1 First Operation

If the system is baked (see section 4.6, "Baking Out" on page 43) the vacuum should be checked. The base pressure should be lower than 10^{-7} mbar (for more details please see section 5.3.3, "Functional Test" on page 49). Check the electrical connections (see section 4.7, "Electronic Units Installation" on page 44).

After storage for a prolonged period please read the start-up procedure given for the CEM after bake out in section 4.6 . **Check the detector voltage given in the Specification Report for your analyzer** (spectra prints: column Udet) with the default detector voltage (menu Analyzer/ Settings). **A monthly check of this parameter (see figure 15, page 29) is recommended.**

5.2 Quick Start

1. Check vacuum conditions.
2. Check sample.
3. Switch on the analyzer supply HSA3500 (see section , on page).
4. Start the acquisition program SpecsLab2.
5. Select the HSA3500 control unit in the menu Analyzer / Settings
6. Switch on the excitation source.
7. Choose the desired slit combination (table 4 on page 23). (A typical slit combination for standard XPS and large samples ~10x10mm is 2:7x20mm / 2:open)
8. Set the Iris aperture to the desired diameter.
9. Set the scan parameter for the region (e.g. figure 31, page 68).
10. Press Validate and then Measure (window: Region Edit).
11. Save the results.
12. Switch off the units.



5.3 Detailed Operation

5.3.1 Slit Setting

There are different settings available with the PHOIBOS Slit Orbit mechanism. To understand the possible slit combination for this analyzer please see section 3.4, "Slit Orbit Mechanism" on page 22.

The optimum setting is reached when entrance slits are aligned along the lens axis, i.e. when the particle number passing through the lens stages and impinging on the hemispherical capacitor entrance slit S1 is a maximum. There is also the correct position for the exit slit S2. Usually there are only two exit slits, therefore physical stops in both directions means that this is also the correct exit slit position. In positioning the feed-through (Figure 12: External Rotary Dial for Positioning page 24) to the slit locations, the rotary dial is internally fixed near to the right value by a physical rest position. Please check the marking at the rotary for the desired combination. Additionally, a check via the viewport for alignment may help. A typical slit combination for standard XPS and large samples (10x10mm) is 2:7x20mm / 2:open.

5.3.2 Detector Operation

For new multiplier (CEM) please read the start-up procedure given for the CEM after bake out in section 4.6. The normal procedure after bake out is increasing the detector voltages in small steps (100V) over a period of 10 min without excitation.

Figure 25: Starting up the detector

Operating conditions:

- A dry-pumped or well-trapped/diffusion-pumped operating environment is desirable. A poor vacuum environment can shorten CEM life or change the operating characteristics.
- A pressure of $1 \cdot 10^{-5}$ mbar or lower is preferred. Higher pressure can result in high background noise due to ion feedback.
- Voltage should be applied to the MCD in small (100 - 200 V) steps.

- For optimal lifetime, operate the detector at the minimum voltage necessary to obtain an usable signal (see section 3.5.3, “Electron Multiplication” on page 27).
- Microchannel plates and Channeltron detectors can be degraded by exposure to various types of hydrocarbon materials which raise the work function of the surface, causing gain degradation.
- If channeltrons are operated at higher temperatures (> 340 K) they can suffer damage. Some channeltrons will lose gain and exhibit a markedly higher detector plateau.

5.3.3 Functional Test

5.3.3.1 XPS (AES/UPS) Operation

To begin, the detector unit should have already been baked out. A silver sample with a size not smaller than 5×14 mm² should have been transferred into the vacuum chamber. The sample has to be cleaned by sputtering. The base pressure should be lower than 10⁻⁷ mbar (10⁻⁵ Pa) to avoid a damage to the detector by sparks. For special reasons, e.g. for depth profiling with noble gas ions, operation up to 10⁻⁶ mbar (10⁻⁴ Pa) is allowed.

Adjust the detector voltage to the value corresponding to the Specification Report of the analyzer or check the actual value (see “The Working Point of the CEM’s” on page 27).

Measure a wide energy XPS spectrum scanning the kinetic energy of the particles from 200 to 1.5 keV, e.g. with an energy step of 500meV and in FixedAnalyzerTransmission mode with pass energy of ~10-20.0 eV (Figure 31: XPS on Silver, Wide Scan (PHOIBOS 150 MCD9), page 67).

Group	Region	Method	Lens	Slit	Mode	Scans	Dwell	Delta	E _{exc}	E _{kin}	E _{pass}	U _{bias}	U _{det}	
300W	Overview 300W, MA13	XPS	MA	2:7x20 12:open	FAT	1	0.01	0.3	1253.6	200 – 1267.4	13	90	1950	

In a first rough test without careful adjustment one should get few 10⁴ cps at the Ag 3d5/2 peak using 100 W Mg K α X-ray source power.

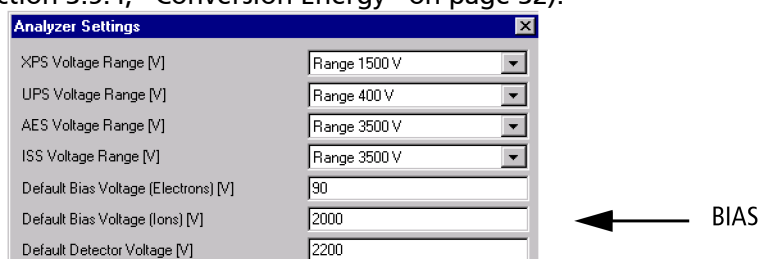
If not, check the photoemission sample current, which should be in the range of 25-40 nA for 100 W Mg K α . Secondly, check the MCD voltage, and the discriminator level of the MCD preamplifier. Also check whether the intensity of the C- and O-peaks are small enough. Otherwise the sample should be sputtered once more.

5.3.3.2 ISS Operation

Please read these instructions carefully. Damage of channeltrons and amplifier are possible if the following is done incorrectly!

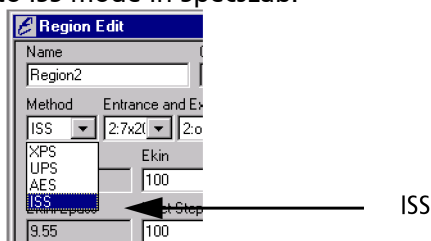
Set the detector voltage to the value corresponding to the Specification Report of the analyzer or check the actual value ("The Working Point of the CEM's" on page 27).

The conversion voltage (BIAS) in the Analyzer Settings window is default set to 2000 (see section 3.5.4, "Conversion Energy" on page 32).

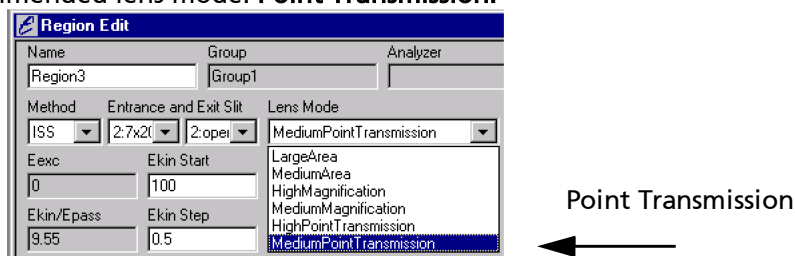


The excitation source should be degassed and run under proper conditions to avoid structures due to possible residual gas inside the sputter line.

Switch the method to ISS mode in SpecsLab.



Recommended lens mode: **Point Transmission**.



Optimize the excitation source with typical sample current of less than 1 μ A. Select the FixedEnergies Analyzer Mode and choose a kinetic energy with measurable counts to maximize intensity with the source deflection tools. Check the function with an overview spectra.

Chapter

6

Troubleshooting

In the section "Possible Problems" (below), a list of possible problems or anomalies and suggestions for their removal is given. It is assumed that the system was calibrated properly and was working according to specifications before one of the following problems occurred. Problems immediately after installation are mostly due to short circuits caused by vibrations inside the analyzer while in transport and may be easily detected by resistance measurements (section 6.1 "Short Circuits" on page 51). Please also note the test described in chapter 8 - "Analyzer Checks" some spectra as well as the measured voltages for diagnostic purposes.

6.1 Short Circuits

Check the resistance of the pins on the HSA 12-pin (figure 33, page 72) and detector (figure 34, page 73) feedthroughs to ground and to each other to rule out short circuits. It is especially important to check for a short circuit between the ground plate and the cathode. The resistivity has to be infinite for all cases, except between the cathode and anode contacts of the CEM. Depending on the type and amount of your channeltrons you will measure few M Ω internal resistance (50-300M Ω per channel or channeltron. Note that the channeltrons are connected in parallel).

6.2 Possible Problems

The following problems may occur during operation of the analyzer system:

- no spectrum
- low intensity
- low resolution
- peaks shifted
- intensity fluctuations
- high background signal
- noisy spectrum
- incorrect area analyzed
- Slit Orbit problems

An arrow (→) after a statement indicates a separate troubleshooting procedure either given here or in another manual.

Possible Cause	perform check, test, or troubleshooting procedure no. →
X-Rays off	check the X-ray source and the sample current
No voltage at detector	check detector voltages
Spectrum definition incorrect	check spectrum definition
Cable connection faulty	check cable connections
Preamplifier box defective	check preamp box → section 9.1.2
Counter device or control unit defective	contact SPECS
No energy sweep voltage	check energy sweep → section and .
Spectrometer voltages incorrect	check spectrometer voltages → section
Improper adjustment of slit orbit	check proper adjustment → section 3.4

Table 5: No Spectrum

possible cause	perform check, test, or troubleshooting procedure no. →
X-ray intensity too low	check whether on Ag sample photo current $\geq 0.2 - 0.3 \text{ nA/W}$
sample is dirty	sputter until C and O peak in the spectrum disappears
sample too rough	remove roughness
incorrect analyzer - sample distance	adjust distance to 40 mm
lens system and HSA out of focus	check lens and HSA electrodes → section 8.4 and voltages section
CEM yield to low	measure detector supply voltages → section
conversion voltage too low	check conversion voltage → section 3.5.4
MCD preamplifier setting changed	check preamp settings → section 3.5.3
some channels of the preamplifier unit defective	check operation of the preamplifier by use of a separated channel mode of the software or → section 9.1.2
magnetic fields	Check whether the amount of the deviation depends on the energy of the measured electrons and on the pass energy (better by UPS). Check the influence of an external permanent magnet near the vacuum chamber.
physically incorrect adjustments like: grids damaged, lenses misaligned,	Open analyzer system and check. Do this only if all other kinds of faults can be excluded.

spheres shifted, MCD shifted.	
improper adjustment of Slit Orbit mechanism	check proper adjustment section 3.4

Table 6: Low Intensity

possible cause	perform check, test, or troubleshooting procedure no. →
chemical peak broadening	sputter cleaning / replace anode
lateral inhomogeneous charging of the sample	use charge compensation by means of electron flood gun
noise or/and ripple on the following voltages: sample, spectrometer voltage U0, detector voltage, electrode voltages of the analyzer	ground sample and check hum and ripple check the ground connection of all the power supplies → section 4.7
magnetic fields in the region of the spheres	measure magnetic field
preamplifier setting changed	check preamp setting → section 3.5.3
lens system and HSA out of focus	check lens and HSA electrodes → section 8.4 and voltages section
detector supply voltage incorrect	check detector supply voltages → section
carbon coating of HSA spheres damaged	open HSA and check - do this only if all other checks are negative
Humming and ripple on the following parts: sample, spectrometer voltages, multiplier voltage, lens voltage	ground sample, check humming and ripple

Table 7: Low Energy Resolution

possible cause	perform check, test, or troubleshooting procedure no. →
workfunction setting incorrect	Check the work function setting, section 7.4 .
sample charging	Check the sample ground connection. Use external electron flood gun to compensate for the charging.
voltages of the HSA (inner and outer sphere) are incorrect	In similar spectra with different pass energies the peaks are shifted. Check has voltages → section 8.4
zero point drift of the spectrometer	check zero point of HSA3500 → section

voltage	
---------	--

Table 8: Peaks Shifted Equally

possible cause	perform check, test, or troubleshooting procedure no. →
incorrect amplification factor	check U ₀ HSA3500 → section 8.2 , section 8.1 , section

Table 9: Peaks Shifted Differently

possible cause	perform check, test, or troubleshooting procedure no. →
malfunction of counter board	→section 9.1.2
one lens electrode not connected	Run a spectrum with high speed. Perform a crosstalk test with all lens tubes. → section 8.4.2
malfunction of the energy sweep generator	check the energy sweep →section , section

Table 10: Intensity Fluctuations

possible cause	perform check, test, or troubleshooting procedure no. →
field emission at the exit slit of the HSA	Background signal independent of E _{kin} with DE=const, rises with small E _{pass} - values and increases in DE/E=const mode. Remove wire-edge from the mesh at the exit slit.
sparks at the anode side of the CEM	background signal is independent of E _{kin} in DE/E=const mode and background to signal ratio decreases with increasing of the pass energy.
incorrect detector preamplifier threshold	→ section 9.1.1
dark current at the isolation parts	check spectra without excitation source 2nd: disconnect the preamplifier and measure again, see Chapter 9

Table 11: High Background Signal

possible cause	perform check, test, or troubleshooting
----------------	---

	procedure no. →
incorrect detector preamplifier threshold	→ section 9.1.1 and section 3.5.3
high noise of the primary source	use a second excitation source,
electric interference	monitor mains voltage, check ground connections → section 4.7

Table 12: Noisy Spectrum

possible cause	perform check, test, or troubleshooting procedure no. →
malfunction of lenses	Check tubus voltages section , ‘Check all analyzer (HSA 3500) voltages” on page . The voltage can be measured either at the 12 pin feedthrough (→ figure 33 on page 72) or at the rear of the HSA3500. Note table 2 on page 16
improper adjustment of the Slit Orbit mechanism	check proper adjustment → section 3.4 , section 4.4
magnetic fields	→section 3.3 and section 4.4

Table 13: Incorrect area was analyzed in all lens modes

Chapter

7

Calibration

Adjustment and calibration of the power supplies has been performed at the factory. Normally no additional work is necessary after installation. The procedures described in this chapter are only necessary for service and fine adjustment.

The typical accuracy of the energy scale depends on the voltage range used (Table , on page). For the 40V range the accuracy is about 3 meV and for the 3500V range about 100 mV without re-calibration. The drifts are due to different temperatures during calibration at the factory and work in the lab. Also, the workfunction of the analyzer can change due to bakeouts; with recalibration the accuracy of the energy scale can be significantly improved (table on page). If energy shifts of well known peaks occur, check the grounding of your sample. There should be no potential difference between the ground of the sample and the ground of the power supply.

Next, check the accuracy of the high voltage ranges (40V, 400V) with a high precision digital voltmeter and the voltage ranges (1500V, 3500V) with an additional high voltage probe (1000:1) section , " " on page). To recalibrate the unit please follow the procedures in figure 27, page 59. Please note the parameters of the modules given in the table on page in section . A description to select a default detector voltage is given in section 9.2 on page 76 and the meaning of the threshold for the preamplifiers is described in section 9.1.1 on page 75.

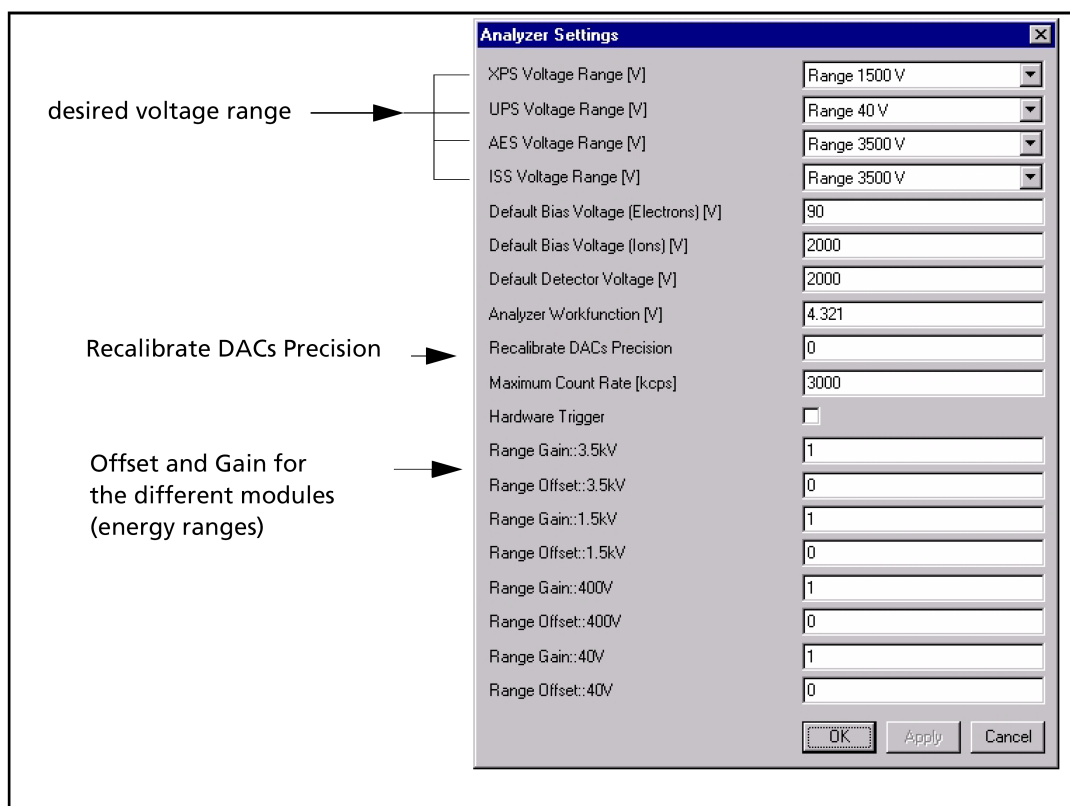


Figure 26: Menu - "Analyzer Settings"

7.1 Complete Calibration Procedure

A complete calibration procedure consists of (see Figure 27: Analyzer Calibration Procedure page 59):

1. Check/set offset and gain with voltmeter, i.e. the calibration of the modules via high precision voltmeter and high voltage probe (section on page)
2. "MCD Calibration", i.e. the estimation of the energy shifts of the single channels for selectable slit/lens combinations (see section 7.3 on page 60), which includes the calibration of the peak position independent of pass energy (see section 8.1 on page 65)

If the achieved values differ substantially from the existing default values or a strong dependence on pass energy is observed for the spectra, this can point out an analyzer malfunction which can not be corrected by the calibration procedure. Please contact SPECS and send some spectra to assist in diagnosis.

3. the estimation of the analyzer workfunction (see section 7.4 on page 61) and fine tuning of the offset for all modules (except 40V) via Fermi edge measurements (Figure 29: Fermi Edge Operation page 62) and optionally

4. Gain calibration with XPS. A calibration check for the gain of the 1500V and 3500V modules see section 7.7 on page 64
5. and in section 8.2.2 , "Check Peak Position" on page 66, verify that the peak position in an XPS fit to the modifications of offset and gain performed in the selected range.

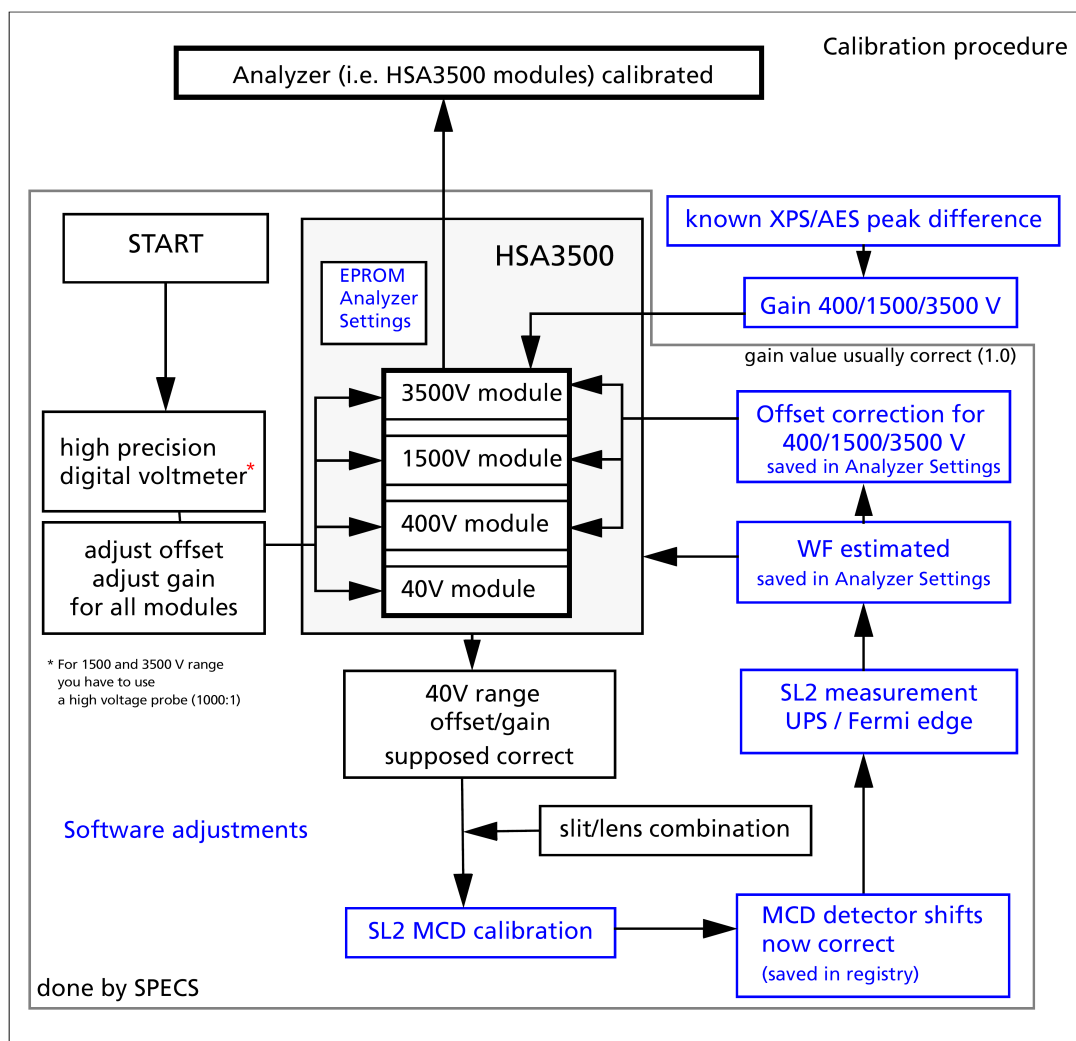


Figure 27: Analyzer Calibration Procedure

7.2 Recalibrate the DAC Precision

Note: the power supply needs time to warm up. A self calibration procedure for the DACs is run every time the power is switched on. You should restart the DAC calibration procedure after a warm up (5-15min, switch off/on or use the field "Recalibrate DAC Precision" in the menu 'Analyzer/Settings' figure 26, page 58). Temperature stability as

well as sample charging or contamination of the surfaces inside the analyzer can influence your calibration.

Perform a manual "Recalibrate DAC" procedure to increase the accuracy to the possible maximum if you have already warmed up the supply for at least 1 hour (e.g. type in a value of 64).

7.3 MCD Calibration

SpecsLab2 software supports a calibration procedure named "MCD Calibration" in the "Tools" menu. (The procedure should be used for the calibration of single channel detectors also.) Because of the energy shift between the single channels for a given kinetic energy (section 3.2, "Hemispherical Analyzer (HSA)" on page 18) and the dependence of the transmitted energy range of α (acceptance angle, see equation 5, page 18) the detector shifts are different for each lens / slit combination. For the most common lens / slit combinations, the values defined by SPECS and implemented in the software give a good approximation. Nevertheless, the most commonly used lens / slit combinations should be calibrated by the customer again to prevent loss of performance.

You can easily check that the values are correct if you display the single channels and compare the energy position of each channel. Second compare two different pass energies. Large differences suggest the need to calibrate this lens / slit combination as described below:

1. Identify a single high intensity peak in your data and set up a region that only measures this peak.
2. Open a separate group with identical regions except pass energy for a single peak within the region (e.g. pass energies 10, 20, 30eV).

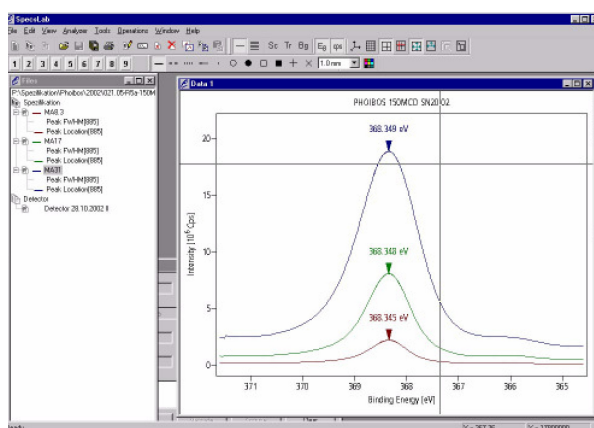
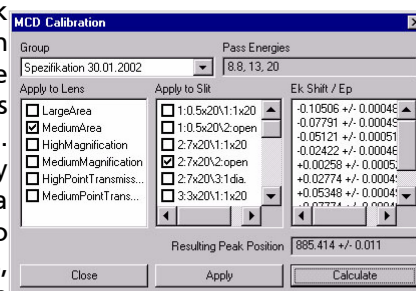


Figure 28: MCD Calibration

3. Measure the regions.
4. Select the tool MCD Calibration in the "Tool" menu.
5. Calculate the 'Ek shift /Ep' by pressing "Calculate" button.

6. Check to see if the differences and the peak position are reasonable. If the calculation failed; check that the peak position can be calculated for all regions. Mostly, regions with low count rates (low pass energies) fail. Move this region into another group and try again (Drag and drop of the region via mouse in the Files window). If you want to check which region is causing the problems, test that each single channel for the region are able to perform the calculation (switch off noisy/wrong channels of the region and check if the peak location with "Tools": "Peak Location" work, if work also MCD calibration will work).
7. Pressing the apply button will apply the calculated shifts to all chosen slit combination. The default values (registry entries) will be overwritten.



7.4 Work Function Calibration with UPS

UV excitation recommended. Please note the comments given in the section 3.6 "Work Function" on page 34. Thermal, aging, or other effects can change the coating of the analyzer and therefore the workfunction over time. Grounding the sample and the power supply influence the work function as well. The default workfunction value can be corrected in the software in the Menu 'Analyzer/Settings', see Figure 26. If you are in doubt about the accuracy of the power supply it is recommended to perform the test / calibration described in section " " on page first. Note the voltage range used while measuring;: 40V, 400V, 1500V or 3500V (see SpecsLab2 menu 'Analyzer/Settings').

1. Warm up the HSA3500 at least 10min. Switch off and on again to reboot and force a startup calibration or manually perform the recalibration of the DACs (section 7.2 on page 59).
2. Set the voltage range for the UPS measurement mode in the Menu 'Analyzer/Settings', see Figure 26, to 40V.
3. Switch to the binding energy scale. Measure the position of the fermi edge (e.g. +0.2 to -0.2 eV binding energy, and select HeI (if He I of course is used) in the dummy source of the Menu 'Analyzer/Settings' Sources selected). (The tool Operation/Fermi Edge in the SpecsLab2 program as well as the cursor and difference cursor cross (black/red cross icon, left/right mouse button) simplifies the procedure.)
4. Calculate the energy difference between expected and measured fermi edge.
5. Note that the high precision measurement with the digital voltmeter for offset and gain are considered as correct and therefore, especially for the 40 V module, no further change for offset and gain is recommended. Once the measured values for this range are correct, than it is possible to estimate the work function.

Correct the default work function value in the Menu 'Analyzer/Settings'.

If the measured difference should be larger than 50meV perform the measurement as described in section , " " on page and change the values. Note that

voltmeter precision and poor grounding of the sample can falsify the calibration!

The work function correction will be applied to all modules!

6. Check the result with new spectra.

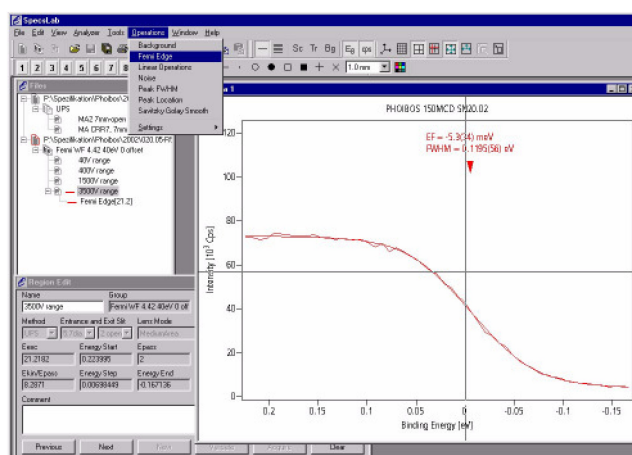


Figure 29: Fermi Edge Operation

Note the advice given in section 7.2 , "Recalibrate the DAC Precision" on page 59
For more detailed information please contact SPECS support.

7.5 Work Function Calibration with XPS

Please note the comments given in section 3.6, "Work Function" on page 34 and in the description in section 7.4.

Instead of the UV excitation (section 7.4), X-ray excitation is used to calibrate the Work Function. Note that you have to select at least the 1500V range (see SpecsLab2 menu 'Analyzer/Settings') in case of Mg/Al Kalpha excitation. This voltage ranges has a lower accuracy than the 40V range (on page).

7.6 Offset Calibration with UPS

UV excitation recommended. Please note the comments given in the paragraph "Work Function" on page 35 and the comments given in section 7.4.

Thermal or other effects can change the accuracy of the offset and gain over time. This can be compensated for with the software. The values for the energy ranges can be changed separately in the Menu 'Analyzer/Settings', see Figure 26. The procedure described in "Work Function Calibration with UPS" on page 58 as well as section , "" on page should be done first if there are any doubts about the supply's accuracy or the Work Function setting.

1. Warm up the HSA3500 at least 10min. Switch off and on again to reboot and force a startup calibration or manually perform the recalibration of the DACs (section 7.2 on page 59).
2. Set the voltage range for the UPS measurement mode in the Menu 'Analyzer/Settings', see Figure 26, to 40V.
3. Switch to the binding energy scale. Measure the position of the fermi edge (e.g. +0.2 to -0.2 eV binding energy, and select He I (if He I of course is used) in the dummy source of the Menu 'Analyzer/Settings' Sources selected). (The tool Operation/Fermi Edge in the SpecsLab2 program as well as the cursor and difference cursor cross (black/red cross icon, left/right mouse button) simplifies the procedure.)
4. Note the comments given in section 7.4 , "Work Function Calibration with UPS" on page 61.
5. Use this 40V range result as the reference point for the other voltage ranges.
6. Select one of the other voltage ranges (400/1500/3500V) in Menu 'Analyzer/Settings' and perform a Fermi edge measurement like for the 40 V range.
7. Compare the result with the 40V range spectra.
8. Calculate the energy difference between expected and measured Fermi edge (the usual difference is less than 0.2 eV, more suggest a hardware error). Set the offset for the selected voltage range (use binding energy values to get the correct polarity for the offset).
9. Set the offset of the used energy module to the calculated value in the menu analyzer settings.
10. Measure again and repeat the procedure for each of the other desired voltage ranges.

The screenshot displays the Spectral software interface for XPS data analysis. The main plot shows the intensity of the PHOBOS 15UMCD S90.02 sample as a function of binding energy. The y-axis represents Intensity in units of 10^5 Cps, ranging from 0 to 120. The x-axis represents Binding Energy in eV, ranging from 0.2 to -0.15. A red curve shows the spectrum, with a vertical red arrow indicating a peak at approximately 5.33 eV. The peak is labeled with its position (EF = -5.33(4) mW) and FWHM (FWHM = 0.1195(50) eV). The software interface includes a menu bar (File, Edit, View, Analyze, Tools), a toolbar, and a sidebar with a tree view of the data. The 'Fence Edge' menu is open, showing options like Background, Linear Operations, Noise, and Settings. The 'Region Edit' panel shows the selected region (PHOBOS 15UMCD S90.02) and its parameters (Energy Start: 27.12462, Energy End: 0.223995, Energy Step: 0.0068444, Energy End: 0.167136).

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For more detailed information please contact SPECS support.

7.7 Gain Calibration with XPS

See section 8.2.1 , "Check Kinetic Energy Scale" on page 66. Run spectra for the gold and the copper peak (use clean samples, sputtered). Measure the distance between the peak maxima of gold and copper. If the value found differs strongly from 848.66 eV please inform SPECS. You can correct the gain (default value = 1) by

$$\text{gain} = \frac{\text{distance}}{848.66 \text{ eV}} \quad (27)$$

Note the default voltage range you have used while measuring with the desired method (see SpecsLab2 menu 'Analyzer/Settings').

Chapter

8

Analyzer Checks

8.1 Independence of Peak Position with Pass Energy

Because of the large number of slit and lens combinations, the energy calibration of the analyzer was not done for each slit combination. SPECS delivers the analyzer with the correct parameters for the common slit combinations. To check the independence of the peak position with pass energy for a desired slit combination please follow the procedure below:

Transfer a silver sample into the system. Warm up your electronics first!

Adjustments:

Excitation	Mg K α
X-ray power	100 W
Slit	desired slit combination, e.g. entrance: largest slit / exit: open
Mode	Medium Area
Binding energy range	365 - 375 eV
Scan	1
Dwell Time	100 msec
Energy step	25 - 30 meV
Pass energy	~ 15eV

Run a spectrum. Set a second pass energy (for the above described slit combination e.g. of about ~5 eV). Repeat the measurement. Compare the spectra. If the voltages are correct, the peak-maxima of both spectra have to have the same energy. The peak positions of both spectra should not differ by more than 100 meV. For a symmetrical peak the peak position is independent of E_{pass} . This is not true for Auger electron peaks since the peaks are intrinsically asymmetrical.

You can also display the single channels for the accumulated spectra. The difference in the peak maxima for each channel indicates the quality of the MCD Calibration (see section 7.3). If the peak energy weakly depends on pass energy, perform the "MCD Calibration" (see section 7.3, page 60). If the calibration does not fix the problem and/or there is a strong dependence please refer to section "" on page . Short circuits or supply failure can cause such behavior. After voltage check please contact SPECS and send some spectra as well as the measured voltages to help diagnosis of the exact problem. Note that there is a weak dependence of the peak energy on beam position at the sample and the size of the illuminated area. Because SPECS calibrates the analyzer with

a broad illuminating X-ray source, additional "MCD Calibration" for the point source may be required.

8.2 Energy Scale Tests with XPS

8.2.1 Check Kinetic Energy Scale

Transfer a gold/copper sample into the system. Clean carefully by ion sputtering. With this sample the main peaks are separated far enough for calibration purposes. The Au 4f_{7/2} and Cu 2p_{3/2} peaks energy difference is 848.66 ± 0.03 eV².

Adjustments:

Excitation	Mg K α
X-ray power	100 W
Slit	entrance: largest slit / exit: open
Mode	Medium Area
Binding energy range	82 - 86 eV for the Au 4f _{7/2} peak 930 - 937 eV for the Cu 2p _{3/2} peak
Dwell Time	100 msec
Scan	1
Energy step	25 - 30 meV
Pass energy	~8 eV

8.2.2 Check Peak Position

Check for proper peak positions corresponding to table 14 on page 67. If the peaks off-set consistently, check if the proper peak position can be achieved by correcting the Work Function, sections 7.4 - "Work Function Calibration with UPS" on page 61, 7.5 - "Work Function Calibration with XPS" on page 62 or 7.7 - "Gain Calibration with XPS" on page 64. If this is not the reason, a complete calibration (as described at the beginning of this chapter) is required or there is a failure in the analyzer or supply (chapter 6, "Troubleshooting" on page 51).

Note: Strong displacement usually caused by poor contact inside the analyzer (see "Connection check for the analyzer electrodes" on page 70) or control unit failure (see "Control Unit Check" on page 61) .

Peak	Mg K α Binding Energy, eV	Al K α Binding Energy, eV
Au 4f _{7/2}	84.00 ± 0.01	83.98 ± 0.02
Ag 3d _{5/2}	368.27 ± 0.01	368.26 ± 0.02
Cu 2p _{3/2}	932.66 ± 0.02	932.67 ± 0.02

² XPS: Binding Energy Calibration of Electron Spectrometers 5 - Re-evaluation of the Reference Energies. M.P.Seah, I.S.Gilman and G. Beamson, Surface and Interface Analysis 26, 642-649 (1998)

Cu L3MM	334.94 ± 0.01	567.96 ± 0.02
----------------	----------------------	----------------------

Table 14: Calibration Binding Energies for non-monochromated Mg K α X-rays

8.3 Specification Check

8.3.1 Survey Spectrum of Silver

The XPS performance of an energy analyzer is usually determined using a silver sample. A clean silver sample is introduced into the vacuum chamber and cleaned by ion sputtering. Use the same settings as in the overview spectrum of silver enclosed with the Specification Report on the analyzer. For example:

Excitation	Mg K α
X-ray power	300 W
Slit	entrance: largest slit / exit: open
Mode	Medium Area
Binding energy range	1000 - 0 eV
Energy step	~300 meV
Dwell Time	100 ms
Pass energy	~13 eV

A typical XPS overview spectrum taken with a PHOIBOS analyzer on silver is shown below (figure 31).

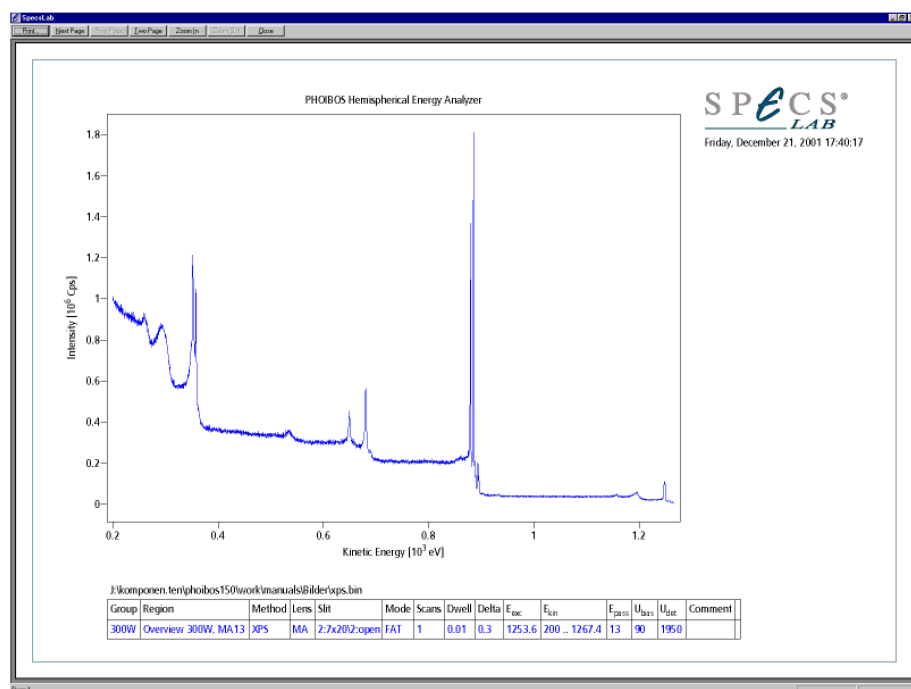


Figure 31: XPS on Silver, Wide Scan (PHOIBOS 150 MCD9)

Parameters for the survey spectrum in figure 33:

Lens mode: **Medium Area**
 Slit: entrance: largest slit / exit: open
 Sample current: 160 nA at 300W Mg K α

8.3.2 Intensity and Resolution

Use the same settings as in the Ag spectra enclosed with the Specification Report on the analyzer.

For example:

Excitation	Mg K α
X-ray power	300 W
Slit	entrance: largest slit / exit: open
Mode	Medium Area
Bin. energy range	378 - 364 eV
Dwell Time	100 msec
No. of scans	1
Step width	25 - 30 meV
Pass energy	8-9 eV

A well resolved Ag 3d doublet is shown in figure 32. This spectrum is typical for a PHOIBOS.

The signal (net intensity, i.e. peak count above background) of the Ag 3d_{5/2} peak is about 85-200 kcps/channel depending on the sample to X-ray source distance. The background in this case is defined as a straight line between the two neighbouring valleys on both sides of the peak. The FWHM of the Ag 3d_{5/2} peak is calculated by measuring the peak width at the half height between the peak maximum and the background.

Note:

Because of considerable spread in the gain of different CEM's, the voltage required for the signal may differ from the specifications (see section 9.2). Pay attention to the detector voltage value in the Specification Report for the analyzer. Monitor the aging of the channeltrons monthly and adjust the default detector voltage.

If the measured spectra and values differ substantially from those in the Specification Report of the analyzer, it may be necessary to sputter the sample again or optimize the sample and X-ray source positions, or a HSA 3500 calibration might be needed. Oxidation of the anode material may also lead to peak broadening.

In many cases it is helpful to know the intensities, signals, and accompanying FWHMs at different pass energies. The measurements described above should be made at pass energies e.g. of 2, 5, 10, 20 and 50eV for largest slit and up to 200 eV for the 1mm slit diameter. The Dwell Time or number of scans should be adapted to the pass energies in such a way that the maximum intensities (in counts not in counts per second) are about the same in every case. Namely, for low pass energies, choose a higher Dwell Time than for high pass energies. This gives comparable counting statistics for all measurements.

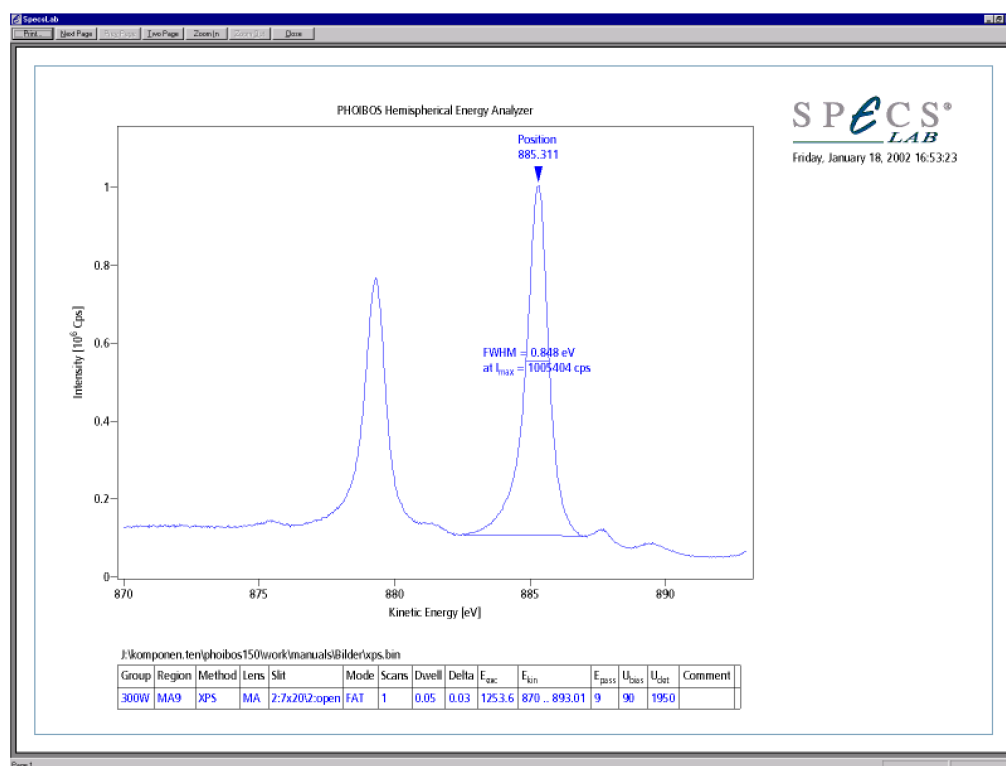


Figure 32: XPS on Silver, Ag 3d (PHOIBOS 150 MCD9)

Parameter for the Ag 3d spectrum in figure 34:

Lens mode: **Medium Area**
 Slit: 2:7 x 20 mm / 2:open
 Sample current: 160 nA 300W Mg K α
 Detector voltage: 1950 V

If no spectrum but a straight line appears after the control unit has been started, either no pulses are arriving at the control unit counter, the counter is defective, or the spectrometer voltage U_0 is missing. The following checks should be made:

1. All cable connections (Figure 2: Connection Scheme page 7) between has 3500 and Detection box PCU 300 the computer and the EC10 Ethernet-Can-Adapter.
2. Control Unit Check, see section
3. Preamplifier, see section 9.1
4. Section 9.2, "Detector Voltage" on page 76.

8.4 Connection Check of the Analyzer Electrodes

In addition to this test, the correct generation of the spectrometer voltages should be checked (see section , " " on page).

8.4.1 Capacitance Check for Electrodes

Check the electrode connection to the electrical feedthrough. For this check a capacitance meter must be used. The best way to check is to measure the capacitances between the housing and all pins of the feedthrough of the analyzer (see Figure 33: Schematics of the 12-pin Analyzer Feedthrough, page 72.). Switch off the HSA 3500 and remove the connector to the HSA electrodes.

! Mind the safety information given at the beginning of this manual!

The capacitances measured on the HSA and lens electrodes under UHV conditions in table 16, and table 17 are for information only. Some differences caused by the meter should be taken into consideration when checking the values. Correct ratios between the values show correct connection to the analyzer parts, the absolute values may differ from the values in the table. Tables of former releases can be found in the documents "Check-PHOIBOSRx.pdf".

PIN	1	2	3	4	5	6	7	8	9	10	11	12
housing	32	147	123	37	121	-	65	71	323	62	170	

Table 15: Capacitance Measurements (pF), PHOIBOS100 R6 or higher

PIN	1	2	3	4	5	6	7	8	9	10	11	12
housing	60	191	139	63	124	-	71	79	396	109	303	

Table 16: Capacitance Measurements (pF), PHOIBOS150 R6 or higher

If the measured capacitances differ substantially from the nominal values please contact SPECS. If the capacitances have nearly the right values, no short circuit inside the spectrometer is likely. If the measured capacitances have the correct values, a missing contact from the HSA 3500 to the analyzer may be the reason for a faulty spectrum (see section 8.4.2 , " on page 71).

8.4.2 Check the Cable Contacts

The connection to the analyzer and the detector are supplied by two multi-pin vacuum feedthroughs, which are designed for high voltages up to 5 kV. Check whether the contacts are in good condition. Since the plug is a movable part which is frequently plugged and unplugged, it can become defective. In most cases, contact failures in the plug are the reason.

- A 12-pin feedthrough on a flange DN38CF, mounted to the lens housing, for all voltages of lens electrodes and capacitor electrodes, which is schematically shown in figure 33, page 72.
- The second 12-pin feedthrough (DN38CF) mounted at the detector flange, for all channeltron voltages and channeltron outputs. The pin assignments is shown in figure 33, page 72.

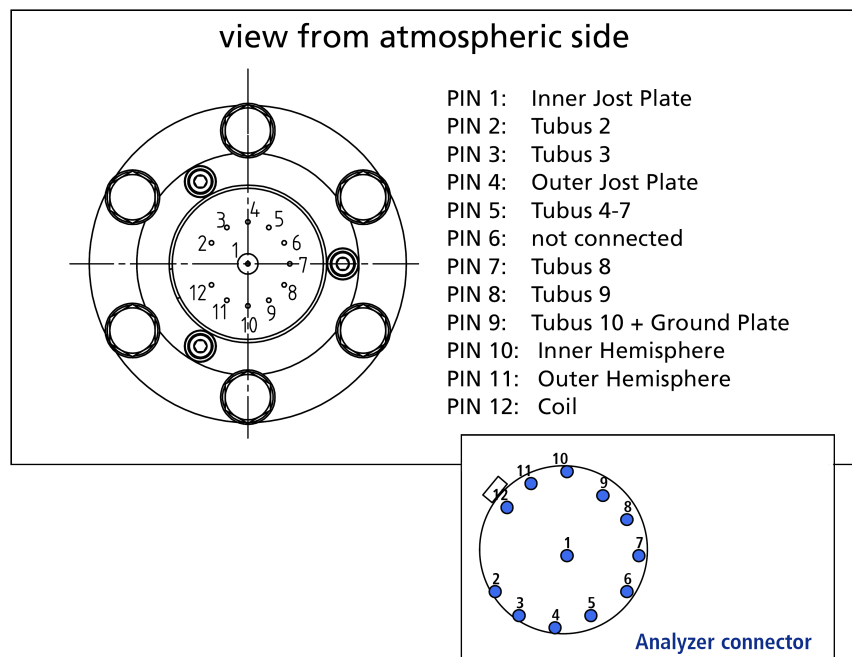


Figure 33: Schematics of the 12-pin Analyzer Feedthrough

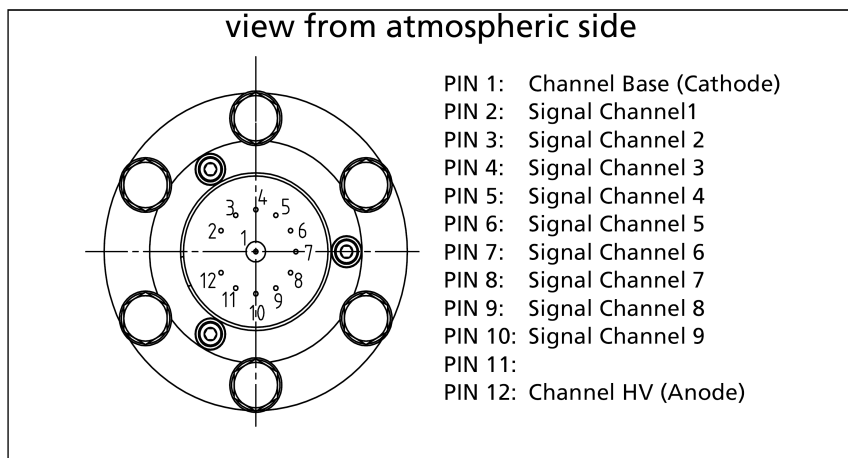


Figure 34: Schematics of the 12 pin Detector Feedthrough

8.5 Check all analyzer voltages

This section has been moved to the HSA3500 Manual.

Chapter

9

Deflector Settings

9.1 Preamplifier

9.1.1 Discriminator

Check the discriminator threshold using the noise of the signal within one spectrum. The square root of the signal (counts! not counts/second) should be equal to the RMS (root mean square) of the noise at this energy (i.e. the value mean signal $\pm 3 \times \text{RMS}$ should include nearly all data points). The noise value without excitation source should be only few counts per second and channel.

Threshold Value:

Use the fixed mode of the acquisition software (see SpecsLab2 manual) to estimate the noise at constant kinetic energy for the acquired signal and compare with the square root of the signal (counts not cps!).

$$\text{Noise} \sim 3 \times \sigma = 3 \times \sqrt{\text{signal} [\text{counts}]} \quad (28)$$

Normally the discriminator threshold does not have to be changed. If necessary, the threshold can be adjusted in the menu 'Analyzer/Settings'. Increasing the threshold level causes a shift of the detector starting point and plateau towards higher voltages, but helps to suppress possible channeltron differences (noise, etc.). Usual threshold values are in the range between 2 and 10mV.

9.1.2 Amplifier Check

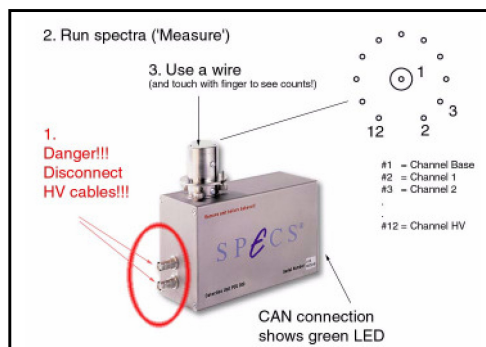


Figure 35: PCU Amplifier Test

Caution

Set the detector voltage to zero and disconnect both HV cables CHANNEL HV and CHANNEL BASE!

! Mind the safety information given at the beginning of this manual!

Disconnect the detector box (preamplifier is inside) from the detector flange. Run a spectrum. Touching each pin of the preamplifier connector with a piece of wire results in a signal. A signal must be observed for each channel. If no signal is observed for some channels, these channels of the MCD preamplifier are not ok. If no signal is observed for all channels or a constant signal is observed, check the preamplifier power (green LED).

Note: If the maximum count rate is achieved the measurement will stop. To avoid this, increase the discriminator level to about 15mV or use a resistor of about 100 Ohm for the check. The default detector threshold is between 2 and 10mV. Do not forget to set the discriminator back to the original or desired values.

9.2 Detector Voltage

Because of a considerable spread in the gain of different multipliers and the threshold level used, the voltage required for the signal for the installed detection system may differ. Pay attention to the detector voltage value in the Specification Report sent with the analyzer. Basically, a working detector voltage is the detector voltage at the beginning of the detector plateau (DetectorVoltageScan, see Figure 15: Detector Sweep, Count rate vs. Voltage, page 30) and has to be checked monthly.

9.3 Noise

Depending on the environment of the analyzer (ground, feedthrough connection status, ...), the handling during transport / installation (bake out, dust, ...) and the history of operation (vacuum, gas load, total amount of accumulated charged particles, etc.) some or all channels may show noise without any excitation. If the "Amplifier Check" on page 80 shows that the amplifier is working correctly, the grounding of the analyzer is ok; by following the advice given in sections 3.5 - "Single and Multichannel Detector (SCD) / (MCD)" on page 25 and in section 9.1.1 - "Discriminator" on page 75, the noisy channels can be suppressed. First, try to set the threshold of the noisy channel to higher value (up to 10 mV). This will also shift the working point for the channeltron (see "The Working Point of the CEM's" on page 27). If this does not help, completely suppress the channel. To continue to work with the analyzer (up to the replace of the defective part) without the signal from the noisy channels:

- Suppress a noisy channel (section 9.3.1)
- Switch off certain channels (section 9.3.2)

9.3.1 Suppress a Noisy Channel

In some cases a channeltron becomes noisy (dust, contamination, overheated, etc.). To avoid disturbing the acquisition of the other channeltrons, increase the threshold level for this channel (Figure 26: Menu - "Analyzer Settings", page 54). Note: You have to select the desired channel first before changing the value to 120mV (maximum) for example.

To reset all channels to the same value select 'all'.

9.3.2 Switch off Certain Channels

To switch off certain channels, it is necessary to modify the registry entry for the HSA3500Analyzer key. For a 150R5c type of analyzer the entry "CounterChannelMask" is located at

"HKEY_LOCAL_MACHINE\SOFTWARE\Specs\Hsa3500Analyzer\Phoibos-Hsa3500\150 R5c"
The necessary type is "binary". Create such a binary entry if it does not exist (Figure 36: CounterChannelMask for MCD9, 1+2+8+9 off, page 78). Please also refer to the description about the registry entries in the SpecsLab2 manual.

The software masks not only the channels, but the detector shifts and channel gains too. In the selection for the channels in the SpecsLab2 analyzer/settings menu the consecutive numbering of the remaining channels start with 1 and end with the number of the available channels.

Note: After any hardware change (i.e. channel replacement), or any registry change (as above), it is recommended to perform an MCD Calibration routine.

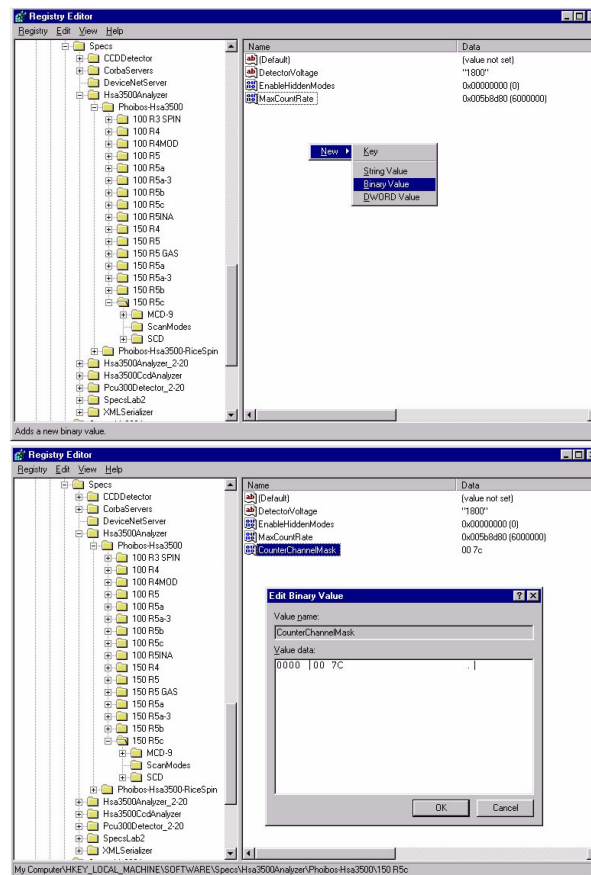


Figure 36: CounterChannelMask for MCD9, 1+2+8+9 off

9.3.2.1 Mask example for MCD9

The *.reg CounterChannelMask entry for a MCD9 type 'PHOIBOS 150 R5c' looks like: Each bit represents one channel. A 9 channel MCD for example has following mask '00000001 11111111' or as hexadecimal '01 FF'. Please note that the first bit from the left is channel number 1 and the last to the right is channel 9 or 5 respectively. Therefore all channels means

[HKEY_LOCAL_MACHINE\SOFTWARE\Specs\Hsa3500Analyzer\Phoibos-Hsa3500\150 R5c]"CounterChannelMask"='01 ff'

To achieve that a 9 channel detector only use 5 channels you have to change to 00000000 01111100 or '00 7C'. The *.reg file for a MCD9 with channel 1,2 and 8,9 switched off looks like following:

[HKEY_LOCAL_MACHINE\SOFTWARE\Specs\Hsa3500Analyzer\Phoibos-Hsa3500\150 R5c]"CounterChannelMask"='00 7C'

9.3.2.2 Mask example for MCD5

The *.reg CounterChannelMask entry for a MCD5 type 'PHOIBOS 100 MCD R5c' looks like:

A 5 channel MCD has following mask '00000000 00011111' or as hexadecimal '00 1F'. Please note that the first '1' from the left is channel number 1 and the last digit on the right side is channel 5 respectively.

Therefore all channel means

```
[HKEY_LOCAL_MACHINE\SOFTWARE\Specs\Hsa3500Analyzer\Phoibos-Hsa3500\100 R5c]
"CounterChannelMask"='01 1F'
```

To achieve that a 5 channel detector only use channels #1,2 and 4 you have to change to 00000000 00011010 or '01 10', i.e.

```
[HKEY_LOCAL_MACHINE\SOFTWARE\Specs\Hsa3500Analyzer\Phoibos-Hsa3500\100 R5c]
"CounterChannelMask"='01 10'
```


Chapter

10

Spare Parts

10.1 Cu Gasket

All CF gaskets are custom made by SPECS. In case you need a replacement please contact SPECS, because it depends on the release of the analyzer. The serial number of the analyzer is marked on the lens flange.

10.2 Multiplier

1. SCD, Single Channel Electron Multiplier replacement for PHOIBOS analyzer
2. MCD, CEM Array replacement
 - 5 channels MCD for PHOIBOS analyzer
 - 9 channels MCD for PHOIBOS analyzer

Please indicate the serial number of your analyzer (flange at the lens housing, e.g. 33.04) in the replacement order. The order of a single channeltrons, a set of channeltrons or a complete replacement of the detector flange including channeltrons is possible.

10.2.1 Channeltron Handling and Storage

A channeltron or a channeltron array (array of 5 or 9 of single channel multipliers, fused together in a precision matrix) is a high gain device for detecting energetic particles such as electron and ions, or radiation. The channeltron consists of a small, curved glass tube. The inside wall is coated with a high resistance material. The resistive material becomes a continuous dynode when a potential is applied between the ends of the tube. It is fabricated from a lead-doped glass. Proper handling is required and the following precautions must be taken.

10.2.1.1 Handling of the Multiplier

- Shipping containers should be opened only under clean, dust-free conditions.
- No physical object should come in contact with the active area of the detector. The channeltron should be handled by its solid borders using clean, decreased tools fabricated from stainless steel, teflon (PTFE) or other UHV compatible materials.

- The channeltrons should be protected from exposure to particle contamination. Particles which become affixed to the plate can be removed by using a single-hair brush and an ionized dry nitrogen gun.

10.2.1.2 Operation of the Multiplier

Microchannel plates and Channeltron detectors can be degraded by exposure to various types of hydrocarbon materials which raise the work function of the surface, causing gain degradation. Operation in a clean vacuum environment of 10^{-5} mbar or better is necessary in order to ensure the long-life characteristics of these devices.

10.2.1.3 Storage of the Multiplier

Due to the hygroscopic nature of the doped lead glass, it is important that the channeltrons are stored properly.

Warning:

The shipping containers are not suitable for storage periods exceeding the delivery time. Upon delivery to the customer's facility, channeltrons must be transferred to a suitable long term storage medium.

- The most effective long term storage condition for the channeltron is a clean (oil free) vacuum.
- A dry box which utilizes an inert gas, such as argon or nitrogen heated above the dew point, is also suitable.
- Desiccator cabinets that utilize silica gel or other solid desiccants to remove moisture have been proven to be **unacceptable**.

10.2.2 Change a Channeltron

A multiplier loses its gain with operating time. It should be changed when a significant degradation in amplification (i.e. intensity) is experienced or the detector voltage limit of the control unit is reached.

10.2.2.1 Removing the Detector Flange

- **Switch off the analyzer supply.**
- Remove both the detector and analyzer connections.
- Vent the system.
- Open the detector flange, let the detector unit down slowly and put it carefully on a table.
(Three springs, pressing the detector assembly to the groundplate may exert some force downward when the screws are released.)

10.2.2.2 Replacing Channeltrons

- Note: Use dry nitrogen only in order to remove dust or lint.
- Loosen the three screws at the top plate and remove the plate.

- Disconnect the cables.
- Note the orientation and alignment of the defective channeltrons.
- Release the screws which are fixing the ceramic rods. Release only one of the adjustment screws. Do not change the other screws, this fixes the position for the channeltrons with respect to the body when rebuilding the assembly (figure 37, page 83).
- Pull back the ceramic rods until the replacement of the channeltron is possible (figure 38, page 83). If the ceramic rod is stuck, try to loosen the screws of each channeltron a bit.
- Remove the channeltron.
- Put the new channeltron in place and rebuild in reverse order.
- Carefully fasten the connections with the screws. Note: A bad contact means noisy channeltron!
- Check all channeltrons for proper mounting.
- Check all electrical connections (Figure 33: Schematics of the 12-pin Analyzer Feedthrough, page 72).

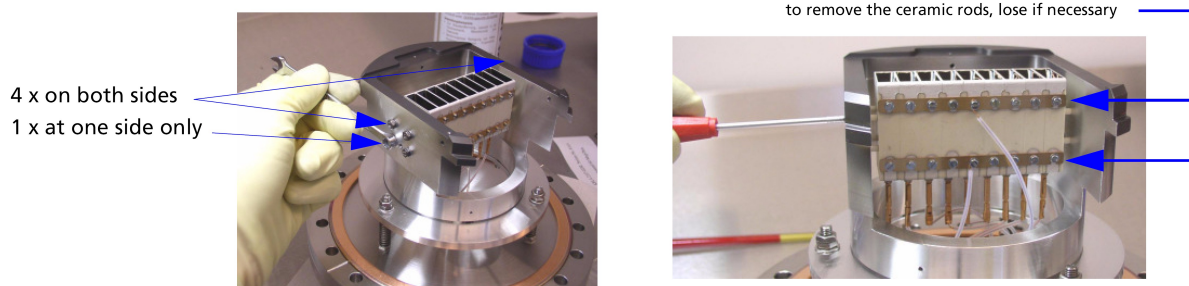


Figure 37: Screws fixing the ceramic rods and adjustment crew

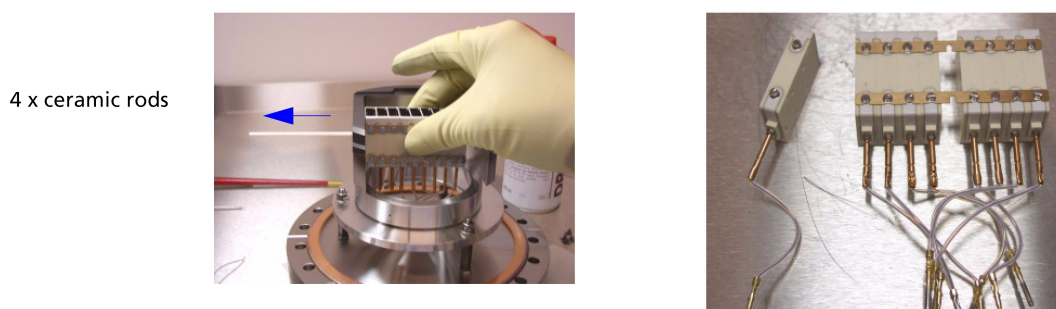


Figure 38: Pull back the Ceramic Rods, Remove the Channeltron

10.2.2.3 Mounting the Detector Flange

- Mount the detector flange in reverse order.
- Check that there is no short circuit for all the pins of the detector supply feed-through to each other and to ground.

- Looking into the opened detector flange from the bottom side you can locate a 'alignment hole' which corresponds to the part at the detector assembly (figure 39).
- Align the pin with the hole in the ground plate. Be sure that the detector is nearly parallel to the ground plate and push the detector carefully into its seat.

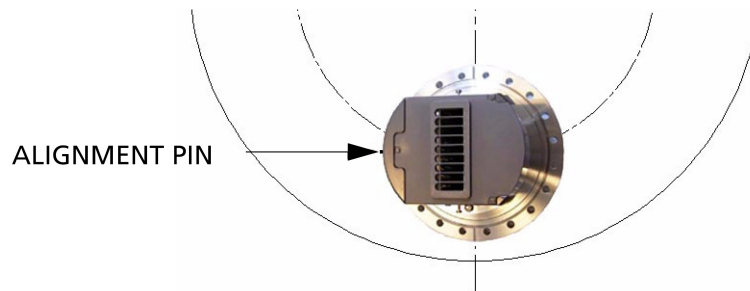


Figure 39: Alignment Pin

- Pump down.
- Detector must be baked out at a vacuum pressure lower than $1 \cdot 10^{-6}$ mbar. It is bakeable up to 200° C
- Check the detector according to section 8.3. (see Figure 15: Detector Sweep, Count rate vs. Voltage page 29); checking the single channels in the acquisition software may be enough.

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Health and Safety Declaration for used Vacuum Equipment and Components

The repair and/or service of vacuum equipment/components can only be carried out if a correctly completed declaration has been submitted for every component.

1. _____ **Description** of **components**
Type: _____ Serial No: _____

2. **Reasons for return** _____

3. Equipment condition

Has the equipment ever come into contact with the following (e.g. gases, liquids, evaporation products, sputtering products ...)

	Yes	No
• toxic substances?	☐	☐
• corrosive substances?	☐	☐
• microbiological substances (incl. sample material)?	☐	☐
• radioactive substances (incl. sample material)?	☐	☐
• ionising particles/radiation (α , β , γ , neutrons, ...)?	☐	☐

Is the equipment free from potentially harmful and hazardous substances? Yes ☐ No ☐

4. Decontamination Procedure

Please list **all harmful substances, gases and by-products** which have come into contact with the vacuum equipment/component during the decontamination methode used.

SUBSTANCE	DECONTAMINATION METHODE

5. Legally Binding Declaration

Organisation: _____
Address: _____
Phone/Fax: _____
Name/Position: _____

I hereby declare that the information supplied on this form is complete and accurate.

Date: _____ **Signature:** _____ **Company stamp**